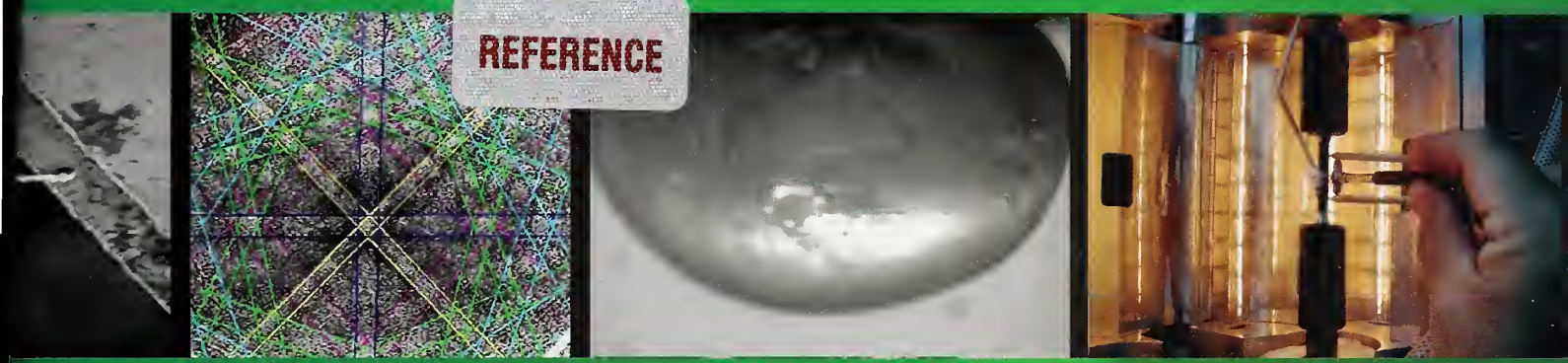
Materials Reliability

FY 2003 Programs and Accomplishments



MSEL

NIST
National Institute of Standards and Technology
Technology Administration
Department of Commerce
NISTIR 7015
November 2003

On the Cover:

Reliability of Modern Materials

Materials Reliability Division research focuses on reliability issues in structures on many scales. Advanced measurement and modeling methods are developed and applied to the characterization of materials properties, along with research on the basic physics and materials science of failure. The images on the cover (from right to left) represent current research focus areas in infrastructure (steel from the World Trade Center being prepared for a high-temperature tensile test), biomaterials (a section of a rat artery inflated to a dome shape during a pressure test), micro/optoelectronics (an electron backscatter diffraction pattern used for elastic strain determination), and nanomaterials (a high magnification image of a carbon nanotube being prepared for electrical testing).

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Commerce for Technology

U.S. Department of Commerce
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Materials Science and Engineering Laboratory

FY 2003 Programs and Accomplishments

Materials Reliability Division

Jim St. Pierre, Acting Chief
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NISTIR 7015
September 2003

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Executive Summary

The Materials Reliability Division's mission is to develop and disseminate measurement methods and standards enhancing the quality and reliability of materials for industry. Our portfolio of research projects has changed again this past year, as we continue to discover and address critical technology gaps that limit the reliable use of materials. The ability to grow in new directions is a testament to our dedicated staff who demonstrate their willingness to take risks in exploring new areas. Currently, our work spans a wide range of materials and with a dimension span that extends from nanometer scale devices to tall buildings, gas pipelines, and bridges. For FY03, we organized our research into the following focus areas:

Materials for Micro- and Optoelectronics

The U.S. microelectronics and related industries continue to face fierce international competition. The International Technology Roadmap for Semiconductors (ITRS) has long recognized the importance of metrology to the advancement of the industry by devoting an entire chapter to the subject. We continue to support the industry through the following projects aimed at improved measurements, modeling, and advanced material science: measurement of mechanical properties and micrometer-scale reliability of thin films; improved understanding of stress voiding (SV), electromigration, and strain in photonic semiconductors; comparison of new theories for thermal flow to actual measurements related to packaging reliability; linking models from interatomic, to nano-, to macro-length scales; and to molecular dynamics simulations. A recent jump in non-self citations in the peer-reviewed journals (22 times in 2001 & 2002) provides evidence of our customers' interest in this important work.

Nanocharacterization

Metrology, the science of measurement, is the foundation of nanotechnology. Manufacturing commercially viable nanoscale products, so widely envisioned in the press, demands vast improvements in our ability to measure material dimensions, characteristics, and structures at the nano-level. The overarching goal of our nine nanocharacterization projects is to develop reliable and accurate measurement techniques for a broad range of materials and material properties at the nanoscale. We are applying a variety of techniques to meet this challenge, such as: Green's function modeling, Brillouin light scattering, carbon nanotube-enhanced atomic force microscopy, atomic force acoustic microscopy, surface acoustic waves spectroscopy (SAWs), and x-ray and electron diffraction methods. We continue to explore synergies between these techniques as well as application of these measurement tools to a wide range of materials: ceramics, polymers, thin films, low-k dielectrics, self-assembled structures, and others.

Biomaterials Metrology

Our ongoing biomaterials projects are aimed at improving health care. These efforts demonstrate the flexibility and professionalism of the division staff who are applying their material science, metrology, and mechanics expertise to efforts that include support for: treatment of pediatric pulmonary hypertension by determining mechanical properties of the pulmonary artery and its constituents; tissue engineering through development of mechanical testing techniques and equipment; cancer research through improved understanding of cellular response to stimuli using Cellular Engineering Micro Systems (CEMS); and cellular-level measurements of mechanical forces and biological phenomena using optical tweezers. Strong and diverse collaborations exist with: University of Colorado Health Sciences, National Jewish Medical Research Center, Children's Hospital of Denver, and Colorado School of Mines. In addition, two R21 proposals were submitted to NIH in June.

Infrastructure

The projects in this group are designed to develop measurement technology for determining a material's characteristics or for characterizing a measurement system. In FY03, we continued to reduce our activities in welding and expanded our activities in lead-free solders. The Charpy SRM program had over 1000 customers in FY03 and shipped more than 1000 copies of our new training video. A three-year test program "International Master Batches" of Charpy impact verification specimens is nearing completion. We continue measuring properties of steels used in the World Trade Center as part of the NIST-led study. A joint proposal (with Metallurgy Division) was submitted to DOT for pipeline research. In addition, the division is assigned specific responsibilities (not yet appropriated) by the congressionally authorized Pipeline Safety Act of 2002 (HR 3609).

Acting Division Chief's Commentary

The Materials Reliability Division continues to expand into new directions in FY03. At the same time, our efforts to maintain competence in infrastructure were clearly validated by our ability to quickly respond to the needs of the World Trade Center Study. The staff are motivated, dedicated, and enthusiastic about their mission and its continuous refinement. Much of our research is concentrated in the NIST Strategic Focus Areas of Nanotechnology, Health Care, and Homeland Security. New staff have been added in both technical and support areas, and staff reassignments to new groups and research areas were completed.

After years of distinguished service to the Division, Fred Fickett retired in May 2003. Under Fred's leadership, the Division moved into new and exciting areas while maintaining its core competences. I served as Acting Chief while a replacement was sought. Due to his outstanding qualifications and experience, Al Clark has been selected as the new Division Chief for MRD, and he will start August 2003.

Jim St. Pierre
Acting Division Chief
Materials Reliability Division

Technical Highlights

The following Technical Highlights section includes expanded descriptions of research projects that have broad applicability and impact. These projects generally continue for several years. The results are the product of the efforts of several individuals. The Technical Highlights include:

- Biomaterials Metrology
- Multiscale Modeling of a Gold Nanoisland in a Million-Atom Copper Crystallite
- Mechanical Reliability at the Nanoscale

Biomaterials Metrology

The materials that make up the cardiovascular system must be well understood given that one in five Americans suffers from cardiovascular disease. The biomaterials metrology program of the Materials Reliability Division aims to develop measurement techniques to support the cardiovascular research community.

Timothy P. Quinn and Elizabeth S. Drexler

Background

Cardiovascular disease is the leading cause of death for Americans — forty-one percent of all deaths are caused by the disease. As of 2000, the most recent year for which we have statistics, 61.8 million Americans suffer from cardiovascular disease [American Heart Association, Heart Disease and Stroke Statistics — 2003]. The goal of the Biomaterials Metrology program is to develop materials metrology methods that support research aimed at understanding and mitigating cardiovascular disease. The project was initiated in the fall of 2001 with the expectation that the unique measurement experience at NIST could contribute to the field.

The technical strategy is to make mechanical property measurement techniques available at three length scales: cellular, cellular plus matrix, and tissue levels. In order to understand the response of cardiovascular tissue to mechanical stimulus, the mechanical properties of the cell must be known, how the cell adheres to the matrix, and, finally, the response of the whole tissue. What makes the problem unique to traditional materials researchers is that the material is not static but is constantly adapting to external stimuli (including the mechanical environment).

The research team in MRD consists of eight core researchers, but the work is leveraged through strong collaborations with the University of Colorado at Boulder, the University of Colorado Health Sciences Center, the University of Colorado at Denver, the National Jewish Medical and Research Center, and the Polymers Division of MSEL.

Funding for the research done in MRD has been acquired from MSEL, from the Advanced Technology Program of NIST, and from the division itself. Grant applications have been made to the National Institutes of Health to further develop the program.

Four projects have been initiated: Pediatric Pulmonary Hypertension, Mechanical Response of Tissue Engineering Constructs, Cellular Level Measurements, and Cellular Engineering Micro Systems (CEMS). The Pediatric Pulmonary Hypertension and Mechanical Response of Tissue Engineering Constructs study the response of whole tissue to mechanical stimuli, while the CEMS project and Cellular Level Measurements project study the response of cells. The CEMS project also is studying how the cells adhere to substrates. Each of the projects is described briefly below.

Pediatric Pulmonary Hypertension

In this disease, the pulmonary arteries offer too great a resistance to flow causing the right side of the heart to increase pressure to maintain the required blood volume. The arteries remodel under the higher pressure, which further increases their resistance, and the heart muscle enlarges to maintain the pressure. Eventually, the heart cannot maintain the required volume of flow and fails.

As part of a larger effort to understand and treat the disease, this project has developed a unique bubble test method to measure the mechanical properties of diseased and normal tissue. Extensive tests using a rat model are underway. Preliminary data are shown in Figure 1.

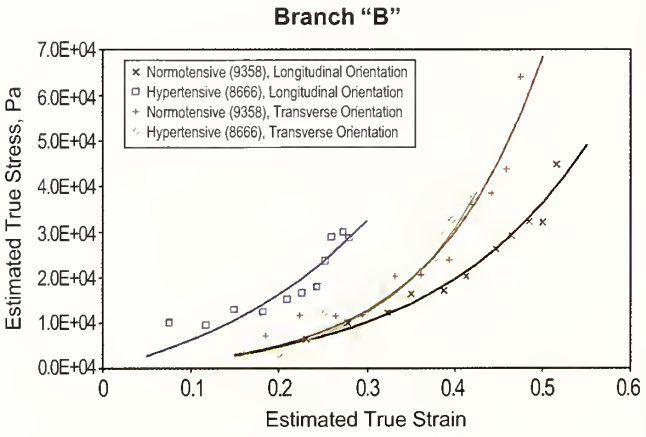

Figure 1: *Preliminary data comparing the longitudinal and transverse stress–strain curves from tests conducted on a normo- and hypertensive rat pulmonary artery.*

Cellular Engineering Micro Systems (CEMS)

One approach to measuring mechanical properties of individual cells and their adherence properties is to make the measurement devices on the same size scale as the cell. Using standard manufacturing techniques developed for Micro-Electro-Mechanical Systems (MEMS), devices have been fabricated that apply and measure the loads and displacements to individual cells. The cells are placed in a physiological environment and made to adhere to substrates deposited directly on the device (Figure 2).

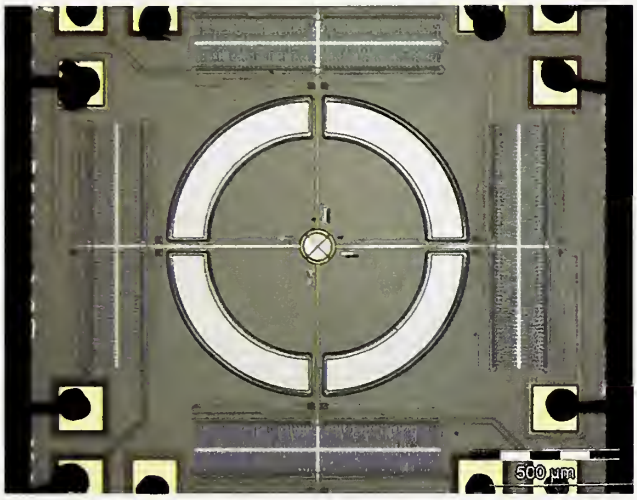

Figure 2: *A packaged CEMS device incorporating comb drives (small white T's at the edge of the device) for the measurement of vascular smooth muscle cells contractile forces. The cell adheres to the device at the small white circle in the center.*

Cellular Level Measurements

This project was initiated in FY02 and aims to develop tools to measure cellular level mechanical properties. This project complements the device development of the CEMS project by introducing calibration techniques. Neutrophils (leukocytes) are being studied because of their importance in their role in inflammatory response of the lung (a factor in pulmonary hypertension).

Mechanical Response of Tissue Engineering Constructs

Diseased or damaged tissue can be replaced with tissue grown on a scaffold designed to promote tissue growth having the proper geometrical, biological, and mechanical properties. The compressive properties of porous polymeric scaffolds have been measured (Figure 3) and modeled in this project. A standard test frame has been designed to grow and evaluate the numerous variables (including stress) that promote the development of the tissue engineering construct.

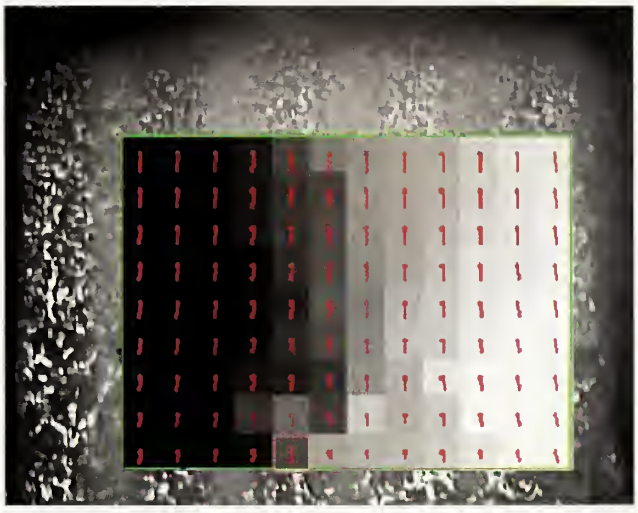

Figure 3: *Image correlation analysis is used to measure the strain in a compression test of a wet polymeric scaffold. The red lines are displacement vectors; the grey levels indicate the relative horizontal displacements.*

For More Information on This Topic

E. Drexler, D. Finch, T. Quinn, A. Slifka (Materials Reliability Division, NIST)

Multiscale Modeling of a Gold Nanoisland in a Million-Atom Copper Crystallite

Modeling and simulation are playing key roles in the discovery and interpretation of nanoscale and atomic-scale phenomena. Multiscale modeling is the most efficient way to include surfaces accessible for measurements in models of atomic-scale phenomena. The brute-force approach would require treating millions or even billions of atoms explicitly. A copper cluster of 1 billion atoms has a side of only 0.23 micrometers, so the desirability of the multiscale approach is clear.

Vinod Tewary and David Read

Our multiscale modeling approach combines molecular dynamics (MD) with a recent extension of Green's function (GF) analysis. We divide the physical system to be analyzed into a core, where the strong, non-linear interactions of interest occur, and an outer core, a shell and a host solid, where the atomic interactions become progressively more linear (termed "harmonic") and the strains decrease to elastic levels. Harmonic behavior can be treated with much less effort, and much less computer power, than nonlinear atomic interactions. In the core, MD is used to maintain the capability to treat strongly nonlinear interactions. The outer core is the region where all the atoms are near their perfect-lattice positions. The core atoms are contained by the shell atoms, which are shared between the MD and GF models, but controlled by the harmonic interactions described by the GF. Outside the shell, the atoms blend into the continuum. Surfaces, with associated strains and displacements, can be placed in the harmonic-lattice or continuum GF regions.

We selected the vacancy in copper as our initial problem to develop and test the method; then we applied the method to a gold nanoisland in a copper matrix. The energy of the vacancy turns out to depend only very weakly on the size of the crystallite considered, so problems in the multiscale approach would have shown up clearly. Even as late as the 1960s, the energy of formation of a vacancy had not yet been calculated accurately. Damask and Dienes, writing in 1963, list values from 0.81 to 1.2 eV. The latest experimental value is given by Wollenberger to be 1.28 ± 0.05 eV. The energy of formation of the vacancy could not be evaluated properly using the pairwise atomic interactions developed during the 1970s and 1980s. However, in the late 1980s and early 1990s, workable many-body potentials were developed. These potentials, fitted to experimental elastic constants, produced accurate vacancy energy values. Here we use a "second moment–tight binding" potential reported by Cleri and Rosato in 1993. It is a very long-range potential, out to fifth neighbors, and parameters were given for mixtures of gold and copper, as well as for the elements individually.

The MD method can evaluate the formation energy of the vacancy; of course, the result depends on the parameters of the MD model used, and the vacancy energy is often considered in fixing the parameters. Both the MD and GF models can calculate the displacements that occur when an atom is removed from a crystallite to create a vacancy. We carried out such calculations with the MD and GF models separately, and they agreed. Then we combined them in the multiscale approach. The multiscale calculation for 216,000 atoms converged after only one iteration. Figure 1 shows the relaxation, and also shows that the strains caused by the vacancy are concentrated in the 100 or so atoms nearest the vacancy. The energy of formation of the vacancy includes these deviations from the perfect-crystal energy.

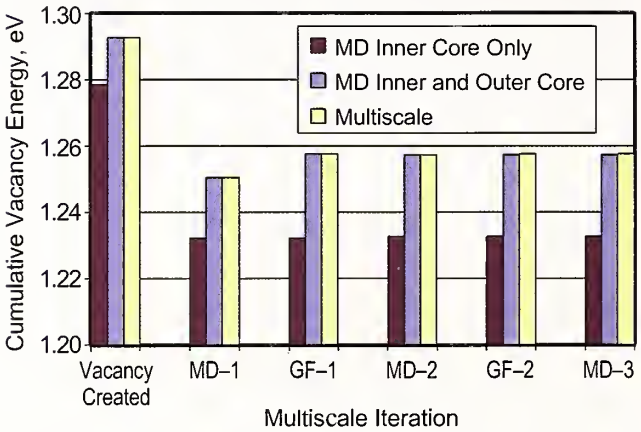

Figure 1: *Multiscale model evaluation of the energy of a vacancy in a copper lattice. The vacancy is created, then the configuration relaxes iteratively to its minimum energy configuration. The MD core contribution to the energy is the largest, because it includes the missing interatomic bonds. Strains in the outer core and the host add to the energy. The latest published experimental result is 1.28 ± 0.05 eV.*

The MD and GF models cooperate closely in the multiscale calculation. The MD model controls the core atoms, and calculates forces on the shared atoms, which are regarded as fixed within the MD calculation. The GF model, in turn, calculates displacements for the shared and outer-core atoms. Because the potential is

many-body, the force on an atom depends on the position of all its neighbors. This dependence is especially pronounced in the fifth neighbor Cleri–Rosato potential used here. The key mathematical "trick" of our multiscale approach is that the many-body force contributions from the host atoms, which are not included in the MD model but are required to get the forces on the shell atoms correct, are evaluated using the equilibrium conditions enforced by the GF model for the outer core, shell, and host regions. As the minimum energy condition is approached, the atomic displacements are smaller so that eventually these forces become constant and the iteration converges.

By replacing 63 atoms within a million-atom copper lattice with gold atoms, we created a gold nanoisland within the copper matrix. Gold's face-centered-cubic (fcc) unit cell is 13 % larger than that of copper; the unknowns were how much the expansion of the gold island would be restrained by the surrounding copper, and what form of the strain and displacement fields around the gold island would be. These turned out to be surprisingly complex. Because the GF model includes the capability to account for a free surface, these strains and displacements are experimentally accessible using, for example, scanned probe microscopy (SPM) or local x-ray diffraction (XRD). This simulation required a larger MD core and shell than the vacancy. We placed 10,156 atoms in the shell and 5,356 in the outer core. Figure 2 shows the initial and final positions of atoms on a section through the gold nanoisland.

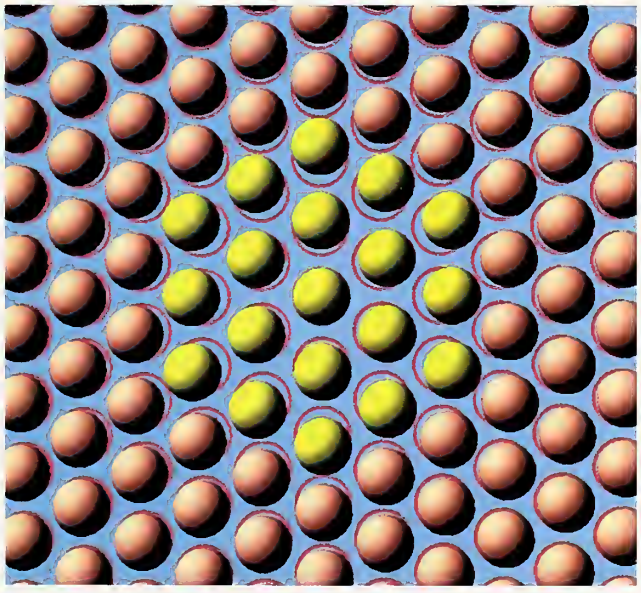

Figure 2: *Section through gold nanoisland in copper after partial relaxation. The red circles indicate the original positions of the copper lattice sites. Note the short range of the displacement field around the nanoisland. The copper nearest-neighbor distance is 2.55 Å.*

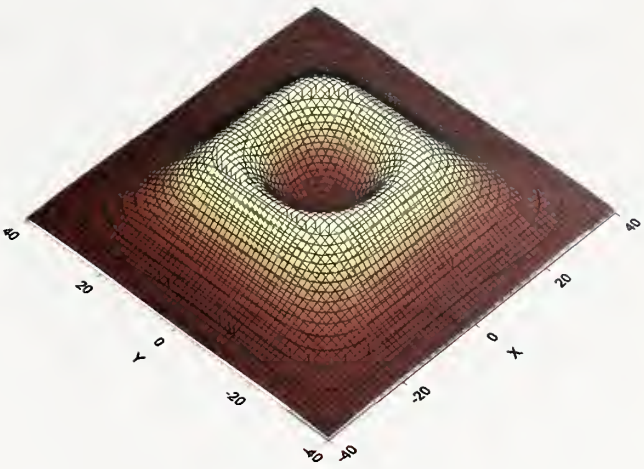

Figure 3: *Out-of-plane surface displacements above an 8 Å-wide gold nanoisland located 38 Å beneath the surface of a million-atom copper lattice. The maximum height is 0.02 Å.*

The gold nanoisland was placed near a surface to calculate surface strains and displacements. Figure 3 shows the out-of-plane displacements. The largest displacement does not occur directly above the nanoisland. Rather, the displacement peaks at the same distance out from the center as the nanoisland is beneath the surface. That the displacement is upwards above the nanoisland is consistent with the larger lattice parameter of gold, compared to copper.

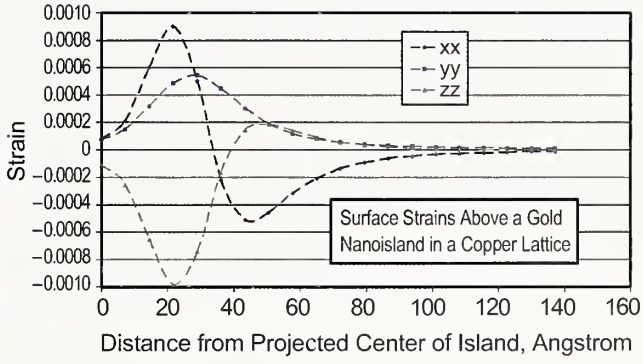

Figure 4: *Surface strains above an 8 Å-wide gold nanoisland located 38 Å beneath the surface of a million-atom copper lattice.*

Figure 4 shows the surface strains above the island. The surface strains above the nanoisland are even more complex than the displacements because of the elastic anisotropy of the copper elastic constants.

For More Information on This Topic

V. Tewary, D.T. Read (Materials Reliability Division, NIST)

Mechanical Reliability at the Nanoscale

Measurement of fundamental material properties at the nanoscale represents a grand challenge for the material science community. In addition to difficulties in manipulating and probing materials and devices on this scale, measurements may need to be made in situ *during fabrication, on materials embedded within layered devices, or within very small spatial regions. To address this complex problem, a suite of characterization tools are being developed and demonstrated that can be used either independently or collectively to determine the relevant nanoscale mechanical and physical properties necessary to ensure device reliability.*

Stephanie A. Hooker

Project Introduction

The emerging field of nanotechnology represents an extremely interesting and challenging opportunity for material scientists. As the fabrication of nanomaterials and nanoscale devices continues to progress at breakneck speed, the need for new test techniques commensurate with this much smaller dimension has become even more evident within the research community. In particular, many material properties must now be revisited in light of the fact that a considerable increase in surface-to-volume ratio is associated with the reduction in material size to the nanoscale, often having a prominent effect on material performance. Historically, fundamental material properties such as elastic modulus have been characterized in bulk specimens using macroscopic, and more recently microscopic, techniques. However, as nanofabrication advances continue, these bulk properties are no longer sufficient to predict performance when devices are fabricated with small critical dimensions. A new array of metrology tools is, therefore, needed to bridge this gap, providing a better understanding of material behavior at the nanoscale and ensuring device reliability — a critical issue for the future commercial success of this technology area.

In response to this challenge, the Materials Reliability Division is currently developing and demonstrating a suite of characterization tools that can be used in a complementary fashion to accurately describe mechanical behavior at the nanoscale. Emphasis is being placed on approaches commensurate with the examination of thin-film structures, especially those with relevant microstructural features below 100 nm (*e.g.*, nanoporosity) and those with one or more critical dimensions in the sub-100 nm range. Desirable measurement attributes for such films include: spatial resolution at or below 20 nm, minimal specimen damage during testing, *in situ* characterization in the final device configuration, and compatibility with a wide range of film/substrate combinations and layered configurations. Of primary interest are mechanical properties, such as Young's modulus and Poisson's ratio, as well as physical properties such as film thickness and density.

Currently, we are conducting studies to compare and contrast a number of different characterization techniques to measure such properties in nanostructured materials. These include atomic force acoustic microscopy (AFAM), surface acoustic wave spectroscopy (SAWS), Brillouin light scattering (BLS), micro- and nano-scale tensile testing, and nanoindentation (in collaboration with the Ceramics Division). By utilizing these tools collectively, a greater body of data can be acquired for a given material set; the certainty of the measured properties can be increased significantly; and the advantages and disadvantages of each technique can be properly explored and compared.

Moreover, powerful techniques based on static and dynamic Green's functions are being developed for mathematical modeling of nanomaterials to further understand mechanical behavior at the nanoscale. In this manner, predicted and measured properties can be linked and compared for a wide range of materials, further increasing the data available to the nanotechnology research community.

FY03 Objectives and Key Accomplishments

Acquiring quantitative mechanical properties over nanometer-scale areas is a primary focus of several Division activities. Over the past few years, an AFM-based technique, referred to as atomic force acoustic microscopy (AFAM), has been under development to address just this issue. AFAM involves vibrating an AFM cantilever at ultrasonic frequencies to excite its mechanical resonance, which then shifts as the tip comes in contact with the material. This shift in frequency is directly related to the material's

elastic properties. AFAM has already been shown to provide quantitative solutions for elastic modulus, and the resulting data have been successfully compared to other techniques such as nanoindentation. However, Young's modulus represents only one piece of the mechanical-property puzzle at the nanoscale. By examining the torsional vibrations of the AFM cantilever, other material properties, such as Poisson's ratio, can also be evaluated. Current research is focused on implementing this approach.

In addition to AFM-based techniques, the development of optical, non-contacting methods for extracting mechanical properties from thin films is also a key objective of the Division's program. In this area, two different techniques are being pursued, namely surface acoustic wave spectroscopy (SAWS) and Brillouin light scattering (BLS). SAWS involves optical generation and detection of a surface acoustic wave traveling along a film surface. This technique can simultaneously evaluate two different mechanical or physical properties (*e.g.*, Young's modulus, thickness, or density). In addition, in FY03, SAWS has been utilized as an important tool to verify nanoindentation data, a critical step in the development of standards for thin film evaluation by that technique.

Brillouin light scattering provides a third non-destructive and non-contacting technique for measuring elastic properties in nanostructured materials. This method involves inelastic scattering of monochromatic incident light by high-frequency, thermally excited acoustic modes within the material. A high-contrast, multi-pass Fabry–Perot interferometer is used to measure shifts in the photon frequencies, which are related to the elastic properties of the material under evaluation. In FY03, this technique was successfully employed to measure Young's modulus and Poisson's ratio for a series of thin films with controlled nanoscale porosity. Anisotropy effects were also observed in the films — information that can be used to predict other behavior, such as dielectric properties.

While each of these techniques can be used independently to measure mechanical properties, considerably more information can be gained by applying the techniques in a complementary fashion. For example, in FY02, efforts were made to correlate modulus results obtained by AFAM, SAWS, and nanoindentation, increasing the certainty of the data. In FY03, this approach was extended to compare and contrast SAWS and BLS data. Such studies will continue in FY04, potentially including other techniques available within the Division such as nanoscale tensile testing of such materials as carbon nanotubes.

In addition to the added measurement certainty afforded by the use of multiple-characterization tools, one measurement approach may simply be insufficient for devices fabricated at the nanoscale. In certain cases, the nanometer-scale spatial resolution provided by AFAM or nanoindentation may prompt the user to employ these techniques with, for example, SAWS to provide a more accurate determination of film density. In other cases, testing of films *in situ* during fabrication may be essential, leading the user to SAWS or BLS where the sample is not contacted or destroyed during the measurement. Finally, measurement of films in an actual device configuration may be necessary for many applications, leading to collective use of several techniques to ensure that the different layers within the device are taken into account.

Collaborations and Partnerships

In a field as multi-disciplinary as nanotechnology, it is critical for research into new measurement tools to be intimately linked with research in classical materials science, device manufacturing, and end-use applications. In FY03, existing collaborations were strengthened and a number of new research partners identified to help ensure that relevant device issues are addressed.

Current collaborators include a number of universities (the University of Colorado at Boulder, the Colorado School of Mines, the University of Denver, the University of Nebraska); foreign institutions (National Physical Laboratory, U.K., Fraunhofer Institute for Nondestructive Testing, Germany); international organizations (VAMAS); industry associations (Sematech); other NIST divisions (Ceramics, Polymers, Magnetics, and Radio-Frequency Technology); and several small businesses (Nanomaterials Research, Synkera Technologies, Tal Materials).

Additional collaborators will be sought to further leverage and grow this program in FY04.

For More Information on This Topic

D. Hurley — *Atomic force acoustic microscopy*; D. Hurley, C. Flannery, V. Tewary — *Surface acoustic wave spectroscopy*; W. Johnson, S. Kim, C. Flannery — *Brillouin light scattering*; D. Read — *Micro- and nanoscale tensile testing* (Materials Reliability Division, NIST); D. Smith — *Nanoindentation* (Ceramics Division, NIST)

Nanocharacterization

The emphasis on nanotechnology around the world and the successful implementation of the National Nanotechnology Initiative in the U.S. are accelerating the development of science and technology at the nanoscale. Nanotechnology is expected to play a key role within the next 10 years in a wide spectrum of industry sectors including manufacturing, information technology, electronics, and healthcare. Novel devices at the micro- and nanoscale will become increasingly important in all of these industries. The ability to measure dimensions, characterize materials, and elucidate structures of new and novel materials at the nanoscale will be critical to the advancement of nanotechnology. In addition, one of the exciting prospects of nanotechnology lies in the ability of molecules or particles, under specific conditions, to self-assemble to form new materials with unusual properties. Successful development of these new materials will require the ability to monitor such processes at the nanoscale in real time. Metrology, the science of measurement, is therefore the foundation of nanotechnology. Standards and reference materials will also provide essential infrastructural support to this emerging technology.

The objective of the program in the NIST Materials Science and Engineering Laboratory (MSEL) is to develop basic measurement metrology at the nanoscale for the determination of bulk and surface material properties and for process monitoring. Measurement methods are being developed for use in conjunction with new instrumentation and calibration artifacts.

The scope of the program encompasses metals, ceramics, and polymers in various forms — particles, thin films, nanotubes, and self-assembled structures — and also includes studies of nanocomposites and liquid-state properties for microfluidics-based fabrication and measurement techniques. Physical properties such as mechanical strength, elastic moduli, friction, stiction, adhesion, and fatigue strength are measured, as well as the size of nanoparticles and the structure and dispersion behavior of nanoparticulate systems. Other properties such as electrical conductivity, thermal conductivity, magnetic properties, electronic properties, and optical properties are also examined. While the program focuses on developing measurement techniques at the nanoscale, proper data interpretation requires fundamental studies in nanomechanics, scaling laws, and imaging techniques.

There are currently ten projects under the program:

- Bridging Length Scales in Theory and Modeling;
- Electrochemical Processing of Nanoscale Materials;
- Metrology for Nanoscale Properties;
- Nanofiller Processing;
- Nanoindentation;
- Nanomechanics and Standards;
- Nanoscale Manufacturing;
- Nanotribology and Surface Properties;
- Particle Metrology and Standards; and
- Physical Properties of Thin Films and Nanostructures.

In many of these individual projects, objectives are directed toward the study of a particular class of materials or material properties, but the underlying theme of the program as a whole is to develop reliable, accurate measurement techniques for a broad range of materials and material properties at the nanoscale. As just one example, four methods for the determination of the elastic properties of thin films — atomic force acoustic microscopy, surface acoustic wave spectroscopy, Brillouin light scattering, and nanoindentation — are being compared using common sets of specimens. This study will lead to a better understanding of the complementary nature of these techniques for measuring nanostructured materials and their combined use to determine supplementary properties such as film thickness or density. Standard reference materials are being developed in collaboration with other National Measurement Institutes around the world for use in the verification of the performance of these instruments.

Contact: Stephanie A. Hooker

Metrology for Nanoscale Properties: Conductive AFM Using Carbon Nanotubes

The latest integrated electrical circuits, as well as microelectical mechanical devices, are dependent on sub-micron features with very high aspect ratios. We are developing a technique using carbon nanotubes mounted to atomic force microscope tips to probe the surface conductivity of these devices with nanometer resolution. The development of these tips also leads to probing high-frequency electronic circuitry and the electrical properties of biological specimens.

Paul Rice

We are mounting carbon nanotubes on the end of conductive atomic force microscope (AFM) tips to probe the surface electrical properties of various samples with nanometer resolution. Using these tips, we are attempting to map the sample surface conductivity and also to probe local electric fields on high-frequency circuits.

Carbon nanotubes have qualities that make them very desirable as AFM probes. They can be micrometers long and only nanometers in diameter allowing for probing devices with deep narrow features such as microelectrical and mechanical systems (MEMS). In addition, there are a variety of types of nanotubes that can have different electrical transport properties. A nanotube can be a ballistic conductor with very low resistance, a metallic conductor, or a semiconductor. The nanotubes are also very durable and can withstand multiple crashes while imaging. For example, we have found that a nanotube we mounted to an AFM tip will spring back after being bent back upon itself and still provide good quality images.

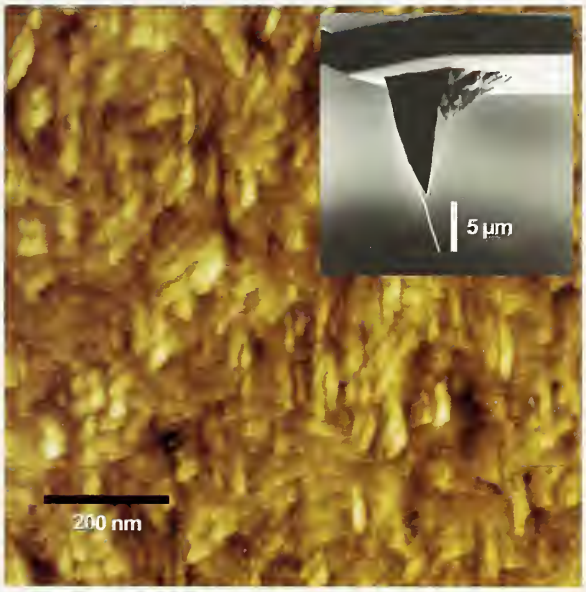

Figure 1: *The image above shows a topographic atomic force microscope image of sputter deposited thin-film niobium. The inset is a SEM micrograph of the AFM tip, fabricated at NIST, which took the AFM image. Attached to the end of the AFM tip is the carbon nanotube that actually probed the sample surface. This image demonstrates the resolution and durability of a carbon nanotube as used for AFM imaging.*

We are currently developing expertise in mounting nanotubes to AFM tips using the SEM. Figure 1 shows an AFM image taken with a nanotube attached to the AFM tip using our in-house technique. The image resolution is comparable to an image taken with a commercial AFM tip. Also, in this application, the nanotube provides extended capabilities for high-aspect-ratio probing, tip durability, and electrical conductance.

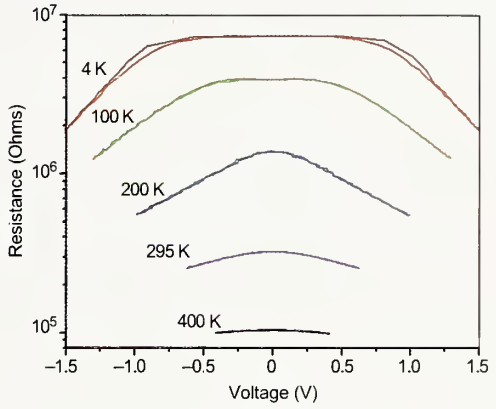

Figure 2: *Resistance versus voltage behavior of a multiwalled nanotube connected to a Au thin-film test structure using electron beam assisted deposition of carbon in the SEM.*

We are also testing the electrical properties of the nanotubes and their subsequent interconnects. Shown in Figure 2 is the resistance versus voltage behavior of a nanotube plus the effects of its interconnects. The resistance response to temperature indicates semiconductive behavior, while the voltage response indicates nonlinear effects possibly due to the interconnects.

Contributors and Collaborators

S.E. Russek (Magnetic Technology Division, NIST); P. Kabos (Radio Frequency Technology Division, NIST); D.S. Finch (Materials Reliability Division, NIST)

Metrology for Nanoscale Properties: Nanoscale Mechanical Properties

We are developing AFM-based techniques to measure nanoscale mechanical properties in situ. Atomic force acoustic microscopy enables quantitative point measurements of elastic modulus as well as images of relative stiffness. The information obtained furthers our understanding of the nanomechanical properties of surfaces and thin-film structures.

Donna Hurley

The accelerating race towards the nanoscale presents a serious challenge for materials characterization. Tools that can assess properties with submicrometer spatial resolution must be developed. Specifically, the need exists for nanoscale mechanical-property information. Knowledge of properties like elastic modulus or stiffness and interfacial quality (defects, adhesion, strain) is critical to successful development of new film materials and nanoscale assemblies. Likewise, such information could help assess integrity or reliability in applications ranging from microelectronics to biotechnology.

To fill this need, we are developing tools that exploit the spatial resolution of atomic force microscopy (AFM). Our approach, called atomic force acoustic microscopy, (AFAM), involves exciting mechanical resonances of the AFM cantilever when in contact with a sample. By comparing spectra for the unknown (test) material and a reference sample with known properties, the indentation modulus M can be measured. The small tip diameter (~20–100 nm) means that we can obtain *in situ* elastic stiffness images with nanoscale spatial resolution.

In FY03, we continued developing AFAM techniques for quantitative measurements. We examined a thin Nb film with two reference samples and two cantilevers. Two analysis methods were used: an analytical model for the dynamics of a uniform rectangular beam, and a finite element method (FEM) that allowed for a variable cantilever cross-section. By averaging the results from both reference materials, values for M were in very good agreement (5 % different) with those obtained by SAW spectroscopy and nanoindentation. For a cantilever that approximated a rectangular beam, the analysis methods yielded very similar values for M. For cantilevers with a nonuniform cross-section, viscoelastic damping had to be included in the FEM analysis. Results from the two cantilevers were equal within measurement uncertainty.

We also examined a hydrogenated silicon carbide film using two reference samples. Values for M with either reference material alone were the same (M=70±3 GPa) within measurement uncertainty as those obtained from SAW spectroscopy and nanoindentation. From these results and those on the Nb film, we concluded that AFAM results are more accurate when the modulus ratio $M_{test}/M_{reference}$ is ~0.9–1.1. We are closely scrutinizing the measurement approach and its contact mechanics to better understand this requirement. Nonetheless, these results demonstrate our ability to reliably and accurately measure elastic properties at the nanoscale.

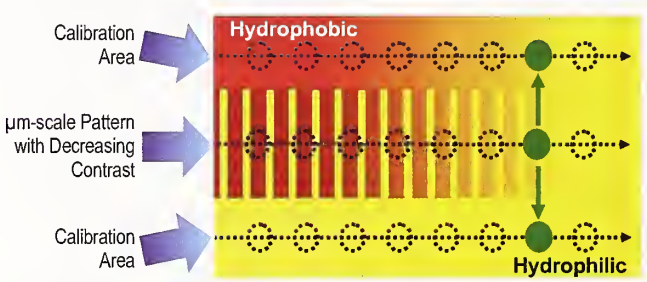

Figure 1: *Specimen for AFAM RH studies. Circles indicate data points along a line scan. Green areas show how minimum contrast to RH sensitivity is determined. (M. Fasolka, NIST)*

Potential relative humidity (RH) effects on AFAM measurements were revealed in experiments on a film of fluorinated silica glass. Values for M correlated roughly with outside RH and increased as the RH increased. We believe that the contact stiffness measurements were affected by a variable water layer between the tip and sample. We are working on laboratory RH control in order to investigate the effect systematically. Samples for these experiments, obtained in a collaboration with Polymers Division, will contain controlled variations in moisture sensitivity as shown in Figure 1. To interpret the experimental results, we are refining our data analysis models to include the effects of the water layer.

The work described above involved measurements of the flexural resonances of the AFM cantilever only. In upcoming months, we will extend our AFAM methods to examine torsional resonances as well. This information will allow us to investigate other nanoscale mechanical properties such as Poisson's ratio or friction.

Contributors and Collaborators

P. Rice (Materials Reliability Division, NIST); A. Kos (Magnetics Division, NIST); M. Fasolka, P. McGuiggan (Polymers Division, NIST); K. Shen, J. Turner (University of Nebraska); N. Jennett (NPL, UK); A. Rar, G. Pharr (University of Tennessee, ORNL); M. Kopycinska–Mueller, U. Rabe, W. Arnold (IZFP, Germany)

Metrology for Nanoscale Properties: Brillouin Light Scattering

A Brillouin-light-scattering facility is being developed for characterizing phonons and magnons at gigahertz frequencies in thin-film materials. The current focus of research is on providing information about the interactions between magnetic modes and thermal phonons, which play a central role in determining the switching times of magnetic-storage devices, spin-valve sensors, and other thin-film magnetic devices.

Ward Johnson and Sudook Kim

Technical Description

Methods that employ Brillouin light scattering (BLS) for the characterization of materials measure the intensity of spectral components of light that is inelastically scattered by acoustic waves (phonons) or spin waves (magnons). Fabry–Perot interferometric techniques are used to acquire accumulated spectra through repeated mechanical sweeping of the etalon spacing.

In this division, research employing BLS is focused primarily on interactions of magnons and phonons in ferromagnetic thin films. This subject is important with respect to maximizing the speed of magnetic-storage devices, spin-valve sensors, and other thin-film magnetic devices because the coupling of spin waves to thermal phonons determines the settling time of the magnetization during a switching event. BLS has the potential of becoming a particularly powerful tool for probing these interactions because it can detect both magnons and phonons at gigahertz frequencies. The aim of our research is to characterize changes in the populations of magnons and phonons induced by ferromagnetic resonant excitation.

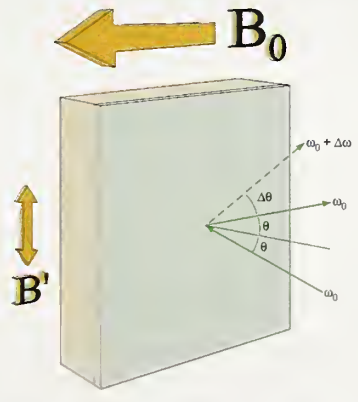

Figure 1: *Scattering of light off waves in a thin film in the presence of static and dynamic magnetic fields.*

Accomplishments

We have implemented techniques for electromagnetically pumping magnons during BLS measurements. As represented in Figure 1, an oscillating magnetic field B′ at gigahertz frequencies is superimposed on a constant magnetic field B_0 to drive spins into uniform precession (excite magnons with a wavevector of zero). Scattered light undergoes a frequency shift $\Delta\omega$ and a change in direction $\Delta\theta$ relative to the specular reflection, as a result of interactions with acoustic or magnetic waves in the film.

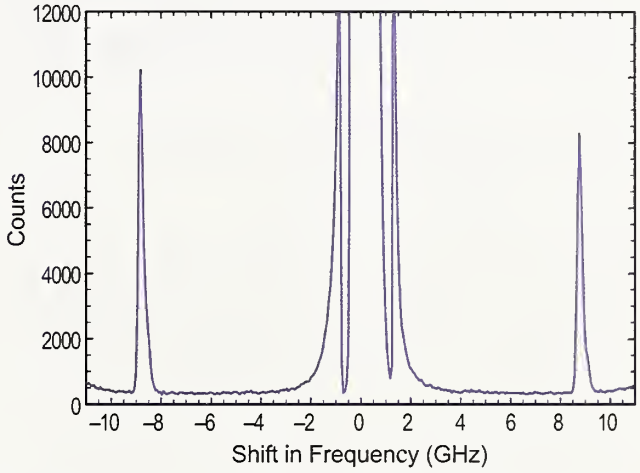

Figure 2: *Backscattered BLS spectrum showing 8.8 GHz pumped magnons in a film of $Ni_{81}Fe_{19}$ (0.9 % Tb).*

This implementation of magnon pumping in a backscattered configuration enables measurements on opaque films, which cannot be studied using forward-scattering techniques. Ongoing measurements are focused on permalloy ($Ni_{81}Fe_{19}$) thin films doped with Tb, which affects magnetic relaxation. An example of a pumped-magnon spectrum from a 50 nm film of $Ni_{81}Fe_{19}$ (0.9 % Tb) is shown in Figure 2. Counts in the range between –2 GHz and 2 GHz arise from a direct reference beam. Strong magnon peaks appear at the 8.8 GHz pumping frequency.

Contributors and Collaborators

P. Kabos (Radio-Frequency Technology Division, NIST); S. Russek (Magnetic Technology Division, NIST)

(This project was partially funded by the National Nanotechnology Initiative.)

Metrology for Nanoscale Properties: X-ray Methods

Macroscopic properties of technologically interesting materials originate from their underlying microstructure. To design and understand improved materials, it is necessary to characterize the microstructure and correlate its changes to the macroscopic properties of interest. We especially focus on x-ray diffraction studies of biomedical, ferroelectric, optoelectronic, photovoltaic, semiconducting, and other materials relevant to the health and microelectronics industries. In particular, studies of microstructural properties, such as strain or stress, crystalline defects, and texture, complement the information obtained by the refinement and determination of short-range and long-range crystal structure in materials.

Thomas A. Siewert and Davor Balzar

In this period, we focused on the studies of shape-memory nanocomposites for biomedical applications and methods of residual-strain determination. Shape-memory polymer-composite materials are capable of recovering relatively large mechanical strains under exposure to moderate temperatures. The typical protocol is to apply a specified initial deformation (called pre-deformation) at an elevated temperature, cool the pre-deformed material under constraint to a lower temperature where the shape is fixed (packaging), and then heat the material to recover the original shape (recovery). The capacity of shape memory polymers to store large strains for deployment in restricted environments has led to their recent use in medical devices. Their primary weakness for medical applications is their inherently low stiffness and low recoverable force levels. The aim of the present research was to examine changes in microstructure from reinforcing the polymer material with ceramics. Such information provides quantitative input into models of packaging and deployment and guides the tailoring of reinforcement type and weight fraction for recovery property optimization. We investigated elastic residual strain from diffraction-line shifts and inhomogeneous defect-related strain from diffraction-line shapes with two different reinforcements (SiC with nominal sizes of 16 nm and 700 nm) during their incorporation into an anmorphous polymer matrix, after subjection of the obtained composites to different degrees of pre-deformation and after subsequent recovery. The results were analyzed for four conditions (pre-deformed at 20 °C, 60 °C, 80 °C, and 100 °C). The stress is compressive and increases proportionally with the pre-deformation temperature. Recovered samples show partial stress relief. This is the first confirmation that the residual strain can be tracked by diffraction in this type of composite, where reinforcement with hard particles (SiC) causes measurable peak shifts in spite of the very soft polymer matrix.

Another area of interest was the refinement of strain- and stress-related parameters in a Rietveld refinement program, as an alternative approach to the traditional methods of strain and stress determination in materials. An advantage of this approach is that all available Bragg reflections are used simultaneously to obtain the strain tensor. However, for successful application in Rietveld refinement, the challenge lies in the accurate modeling of strain and stress dependence on the crystallographic direction and the ability to handle arbitrary crystal symmetry. A recently published model (N.C. Popa and D. Balzar, *J. Appl. Cryst.*, **34** (2001) 187) allows for accurate modeling of diffraction-line shifts in Rietveld-refinement for all Laue symmetries without making Voigt or Reuss approximations. To test this model, we conducted the neutron time-of-flight (TOF) measurements on the SMARTS instrument at Los Alamos Neutron Science Center. The neutron TOF diffraction measurements yield the whole diffraction pattern for Rietveld refinement at every sample orientation. Multiple data banks of SMARTS yielded *hkl*-dependent strains for different crystalline and sample directions, which provided us with the required data to fit the spherical harmonics coefficients describing the strain state. The measurements were collected on two samples of uranium (undeformed and plastically deformed), which allowed us to determine changes in the residual strain state after plastic deformation. A particular advantage of the determination of the strain tensor through Rietveld refinement is that multiphase components and low crystalline symmetry systems with overlapping reflections can be reliably analyzed. An orthorhombic crystalline structure is one of the reasons why we selected pure uranium for the measurements. This research will further our ability to obtain complete strain, stress, and texture information about materials of arbitrary crystalline and sample symmetries.

Contributors and Collaborators

G. Stefanic (Institute Ruder Boskovic, Zagreb, Croatia); N.C. Popa (Dubna, Russia); M.A. Matin (University of Denver); K. Gall; M. Dunn; A.M. Hermann (University of Colorado); S. Vogel (Los Alamos National Laboratory)

Physical Properties of Thin Films and Nanostructures: Multilayer Ceramic Components

A wide range of chemical compositions is now being produced commercially as nanopowders. The availability of ceramic materials in this size classification is particularly important for the electronic component industry. Nanopowders represent an enabling technology for achieving significant decreases in ceramic layer thickness, minimizing the size and prevalence of processing-induced defects, and boosting the overall performance of components ranging from capacitors to piezoelectric actuators. Quantification of these performance enhancements and their association with nanometer-scale microstructure control is a key objective within the Materials Reliability Division.

Stephanie A. Hooker

Surface-mount electronic components are widely used to accomplish both active and passive functions in state-of-the-art circuitry. Examples include capacitors, resistors, surge suppression devices, and temperature sensors. Recently, the multilayer fabrication process used to produce these electronic components has been employed to prepare miniature actuators. These piezoelectric ceramic devices (see Figure 1) provide precision, nanometer-scale motion in a small package and require very low power input, making them highly desirable for use in adaptive structural elements, biomedical pumps, and optical translation devices.

Figure 1: *Photograph & SEM image of typical mutlilayer components produced by Synkera Technologies.*

However, long-term cyclic fatigue, defined as a recoverable or non-recoverable change in polarization or displacement over time, is a recognized problem in piezoelectric actuators. One method to minimize actuator fatigue is to reduce both the size and prevalence of processing-induced defects within the ceramic layers, as these defects can grow rapidly during long-term exposure to electric fields. The emerging prevalence of submicrometer and, more recently, nano-sized ceramic powders offers the opportunity to achieve a high level of defect control in these components, significantly extending their operational lifetime and improving their overall electromechanical performance.

In FY03, a program was initiated to develop specialized test platforms and methodologies to systematically quantify the performance advantages of *nanometer-scale* microstructure control for multilayer actuator components under real-world operational conditions. This new capability combines measurements of dielectric properties, ferroelectric properties, and physical displacement with the simultaneous application of a range of external stimuli including both electrical and mechanical influences. Variables include the input waveform characteristics, loading and/or pre-stress condition, and method of attachment within the system.

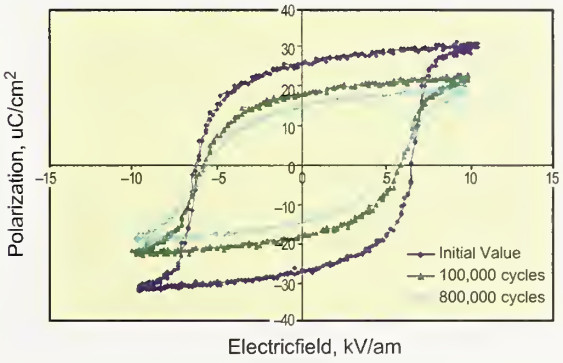

Figure 2: *Polarization degradation for one actuator device as a function of cyclic operation over time.*

To date, a series of microstructure-controlled multilayer actuators has been fabricated by our collaborator, Synkera Technologies, and fully characterized within the Materials Reliability Division of MSEL. Significant enhancements in polarization, electro-mechanical coupling, and resistance to short-term fatigue (see Figure 2) have been identified and correlated with microstructure evolution and grain growth. These results have clarified the importance of understanding the sintering characteristics of nanopowder materials and the relationship between initial precursor size, fired microstructure, and device performance. A more extensive follow-on investigation is currently underway, addressing alternative materials, additional process variables, and more comprehensive accelerated long-term fatigue testing.

Contributors and Collaborators

C. Kostelecky, D. Deininger, K. Womer (Synkera Technologies)

Physical Properties of Thin Films and Nanostructures: Mechanical Properties of Thin Films

We develop and apply noncontact, nondestructive tools to measure thin-film mechanical properties such as elastic modulus and residual stress. Optical methods are used to determine the frequency dependence of the velocity of surface acoustic waves. The information obtained can assist in developing new film materials. It is also valuable for predicting the reliability and performance of thin-film components.

Donna Hurley and Vinod Tewary

To successfully develop a new thin-film material, knowledge of its mechanical properties is often needed: to estimate surface residual stresses, to predict reliability or performance, or to assess film-substrate adhesion. Yet methods are generally limited to destructive tests or even "try it and see." Also, film properties usually cannot be extrapolated from those of bulk samples — if they exist.

We are developing nondestructive methods to quantify thin-film properties. We aim to relate physical properties like elastic moduli, residual stress, or adhesion to film microstructure, quality or performance. The approach exploits the behavior of surface acoustic waves (SAWs). The SAW velocity varies with frequency and depends on the film and substrate properties (Young's modulus E, Poisson ratio, density, and film thickness). We use laser ultrasonic methods to generate and detect SAWs over a broad frequency range. The SAW velocity-*versus*-frequency data are interpreted with a Green's function method to determine the film properties.

In FY03, we implemented a second SAW apparatus. Silicon and other materials are not opaque to infrared light and thus are ill-suited to the infrared detector in the original apparatus. The new apparatus uses a green laser in its interferometer, extending capabilities to a wider range of materials. In addition, a pulsed ultraviolet laser improves generation efficiency in polymer and other films. The apparatus is quite user-friendly, increasing accessibility to postdocs and visitors. This setup frees up our first apparatus for speculative experiments requiring more frequent reconfiguring of optical components.

Analysis capabilities were improved by extending the anisotropic Green's function inversion software to two layers. TiN films were analyzed with three different models: one elastically isotropic layer, one anisotropic layer, and two layers (isotropic TiN over hexagonal Ti). SEM images such as that in Figure 1 show a thin Ti interlayer, but, until now, we could not model two separate

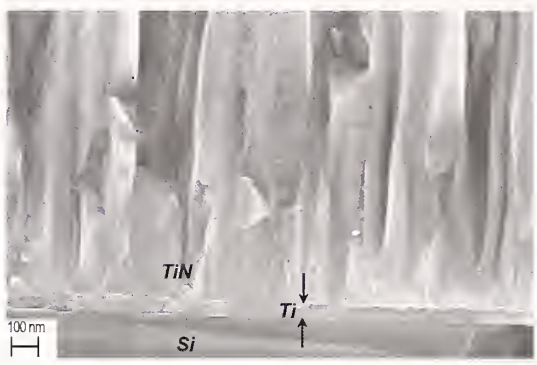

Figure 1: *SEM micrograph of TiN film showing preferential crystallite orientation and adhesive Ti interlayer (R. Geiss, NIST).*

layers. Of the three models, the velocities predicted by the two-layer model agreed best with experimental values. The nanoindentation and two-layer SAW values for E were virtually identical for the thickest film. Discrepancies in other films may be due to acoustoelastic effects or indentation substrate effects.

The two-layer model proved valuable in other cases. In a mini round-robin project for VAMAS TWA 22 begun this year, we examined oxide films (TiO_2 and SiO_2) on glass. SAW measurements on these transparent samples required the addition of a Cr topcoat. The Cr properties were fixed in the two-layer model using results from a sample with a single Cr layer. Preliminary nanoindentation results were in excellent agreement with the SAW results. Also, low-k dielectric films containing secondary capping layers were evaluated with this model (described in detail on the next page of this report).

Although nanoindentation is in widespread use, absolute validation by independent means is needed. The VAMAS project above is one example of our efforts to carefully compare SAW and nanoindentation results. We also performed similar comparisons on a Nb film. In that case, SAW data were obtained at both NIST and another laboratory on the same sample. By combining SAW and nanoindentation information so as to exploit the different sensitivity of each method to different parameters, we obtained a more consistent and complete set of values than possible with either method alone. We will pursue these and related issues (*e.g.*, ultimate uncertainty) during the visit of a guest scientist in late FY03.

Contributors and Collaborators

C. Flannery (Materials Reliability Division, NIST); D. Smith (Ceramics Division, NIST); N. Jennett (NPL, UK); U. Beck (BAM, Germany); D. Fei (Caterpillar)

Physical Properties of Thin Films and Nanostructures: Mechanical Properties of Low-κ Dielectric Films

Although crucial to the microelectronics industry, implementation of low-dielectric-constant (low-κ) films presents serious materials challenges. We apply different optical methods to these films to evaluate their critical properties: density/porosity, Young's modulus, and Poisson's ratio. The techniques provide complementary data and allow measurement of properties that are otherwise difficult to obtain.

Colm Flannery and Donna Hurley

Miniaturization in the microelectronics industry requires lowering the resistance-capacitance (RC) factor in order to achieve faster device switching. This requires replacing standard silica dielectrics with materials of a lower dielectric constant (κ). The most promising way to achieve this is to introduce nanometer-sized pores that reduce κ as porosity increases. An unwanted by-product is a drastic reduction of mechanical properties. The very low Young's modulus of porous low-κ films lessens the chance of their surviving the microelectronic chemical-mechanical polishing process and affects process reliability. Accurate values of film modulus must therefore be known. However, few tools are available to characterize such relevant thin-film properties and their dependence on porosity. Nanoindentation, a standard industry technique, is challenging on such soft films and generally tends to overestimate stiffness significantly. As an alternative, we are developing optical techniques to enable non-contact property measurement.

Brillouin light scattering (BLS) relies on a photon–phonon collision process where incident photons collide with ambient thermal phonons in the material. The frequency shift of the scattered photons, detected by a scanning Fabry–Perot interferometer, is characteristic of the acoustic phonon modes in the film. With BLS, we examined a series of methylsilsesquioxane (MSSQ) polymer films of varying porosity. We could detect both longitudinal and surface acoustic wave modes and, thus, could determine values for both Poisson's ratio ν and Young's modulus *E*. Figure 1, which plots the values of ν obtained in these films *vs.* porosity, reveals that ν decreases as porosity increases. This is important since the dependence of n on porosity is not well understood. Very few techniques have been able to provide experimental data to address this issue. BLS appears to be a very promising tool with which to explore this area.

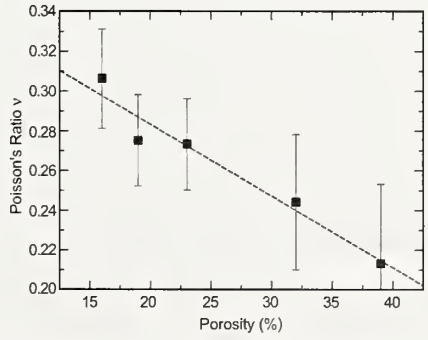

Figure 1: *Poisson's ratio vs. porosity for MSSQ films.*

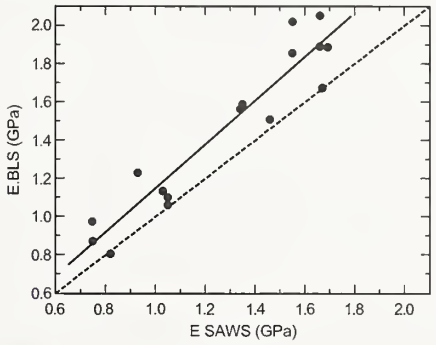

Figure 2: *Young's modulus determined by SAWS and BLS.*

In addition to BLS, frequency-dependent dispersion of laser-generated surface acoustic waves can yield accurate measurements of the density and Young's modulus of thin films. In FY03, we implemented a new SAWS apparatus optimized to inspect thin films on silicon and paid special attention to developing a robust data analysis procedure. Figure 2 compares values of *E* obtained by BLS and SAWS for a range of low-κ films. The results show a clear correlation but with a systematic difference between the techniques of 10–15 %. Reasons for this small discrepancy are being investigated. (SAWS density values have also been verified by x-ray reflectivity measurements).

We are also characterizing two-layer samples (low-κ film with a thin capping layer), interpreted using our newly-developed two-layer Green's function analysis code. Preliminary results show that SAWS is sensitive to the presence of capping layers as thin as 50 nm. Work in the coming year will concentrate on extending SAWS and BLS measurements to multilayer samples, analyzing measurement uncertainties, and developing a standard SAW thin-film data analysis procedure.

Contributors and Collaborators

S. Kim, V. Tewary (Materials Reliability Division, NIST); W. Wu (Polymers Division, NIST); J. Wetzel (Tokyo Electron America)

Physical Properties of Thin Films and Nanostructures: Green's Function Methods

We have developed computationally efficient techniques and computer codes for calculating elastic Green's functions with virtual forces and boundary-element method for application to a variety of elastostatic and elastodynamic problems in advanced materials systems such as semiconductor and metal substrates containing thin layers and nanoinclusions. Many of these codes are available from our library of Green's functions at the NIST website on the Internet.

Vinod Tewary and Bo Yang

Technical Description

Mathematical modeling of the elastic response of anisotropic materials is required for interpretation and design of experiments leading to industrial applications of advanced materials system such as metals and semiconductors with multilayer coatings or quantum nano-inclusions. The elastodynamic and elastostatic Green's functions (GFs) are used for modeling the propagation of elastic waves, and for calculations of stresses and strains.

We are developing a new defect GF method for elastic response of composite material systems such as thin layers on a substrate or nanoinclusions in a host solid. The defect GF method is well known in discrete lattice statics but is new in continuum mechanics. The traditional technique for solving elastic problems in composite materials consists of solving the equations of elastic equilibrium in different regions and then coupling them with appropriate continuity conditions. We define the defect GF that gives the response of the composite system and seems to be computationally more convenient. The defect GF, denoted by $\mathbf{G}^*$, is related to $\mathbf{G}$; the GF of the host solid through the Dyson integral equation as follows:

$$\mathbf{G}^* = \mathbf{L}^{*-1} = (\mathbf{L} - \Delta\mathbf{L})^{-1} = \mathbf{G} + \mathbf{G}\,\Delta\mathbf{L}\,\mathbf{G}^*,$$

where $\mathbf{L}^*$ and $\mathbf{L}$ denote respectively the Christoffel operators of the composite and the host solid, and $\Delta\mathbf{L}$ denotes the change in $\mathbf{L}$ in different regions.

We have extended our earlier GF method for calculating surface acoustic wave (SAW) velocities to multilayered substrates to account for imperfect bonding between the film A and the substrate B by modifying the continuity conditions at the interface as follows:

$$\mathbf{u}_A = \square\,\mathbf{u}_B,$$

where $\mathbf{u}$ denotes the displacement at the interface and $\square$ is the mismatch tensor (assumed to be diagonal), and the subscripts A and B refer to solids A and B.

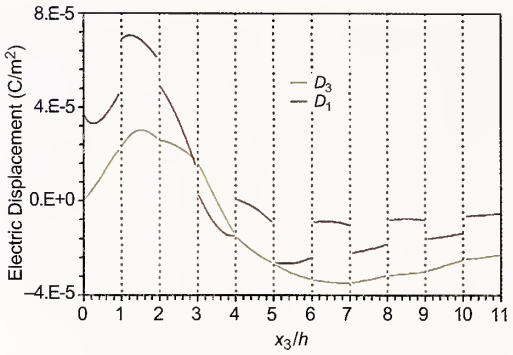

Figure 1: *Response to a unit point force normal to the surface of a multilayered piezoelectric solid (InN/AlN). Vertical lines indicate interfaces between adjacent layers.*

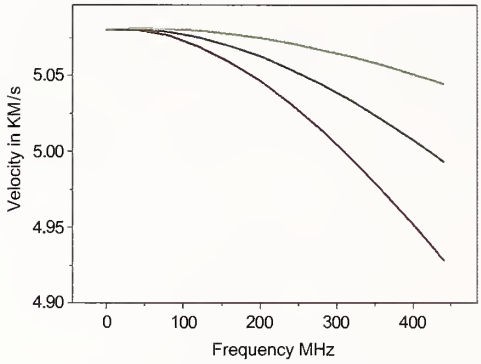

Figure 2: *SAW velocity in TiN for $\square_i = 0.75$ (upper curve), $\square_i = 1$ (middle curve) and $\square_i = 1.25$ (lower curve).*

Accomplishments

We have applied our methods to study the formation of surface quantum dots, static response of an anisotropic multilayered piezoelectric structure, steady-state motion of defects in semi-infinite solids and bimaterials, elastic fields due to inclusions in anisotropic half space, and SAW propagation in a TiN film on silicon with a defective interfacial bonding. Computer codes for GF and boundary-element method for a variety of material systems are available for download from our Library of Green's functions at **www.ctcms.nist.gov/gf**. Finally, we have actively collaborated with the formation of the Digital Library of Green's functions in the National Science Digital Library project that was formally launched in December 2002 by the NSF.

Contributors and Collaborators

D. Hurley, R.R. Keller (Materials Reliability Division, NIST); L. Bartlolo (Kent State University); A. Powell (M.I.T.); E. Pan (Akron University)

Physical Properties of Thin Films and Nanostructures: Molecular Electronic Interconnects

Electronic devices based on large molecular assemblages are being rapidly developed. Further progress is limited by the quality of the contacts joining these devices together. We are exploring various techniques to manipulate and join nanotubes to substrates. We are then developing techniques to probe the subsequent mechanical and electrical characteristics of these nanometer-scale connections.

Paul Rice

Molecular electronic devices such as carbon nanotube transistors have the potential to revolutionize the electronics industry by reducing circuit sizes dramatically and by increasing operational speed due to the inherent properties of the materials. Carbon nanotubes are currently one of the basic building blocks for molecular electronics. The electrical and thermal properties of these molecules theoretically solve numerous materials problems. However, even though, for example, their internal electrical conductivity is very high, the contact resistance between nanotubes is also quite high, limiting the current that can be passed from tube to tube. Using manipulation and fabrication techniques developed within the Division, we are exploring methods to characterize these interconnects.

Figure 1 shows an image of a popular technique called electron beam deposition (EBD) that is used for connecting nanotubes to substrates. In the scanning electron microscope (SEM), the beam is held on a spot where a connection is desired. The beam has enough energy to split hydrocarbon molecules, common contaminants in most SEMs. The resultant active carbon condenses on the nanotube and substrate to form a strong bond or weld.

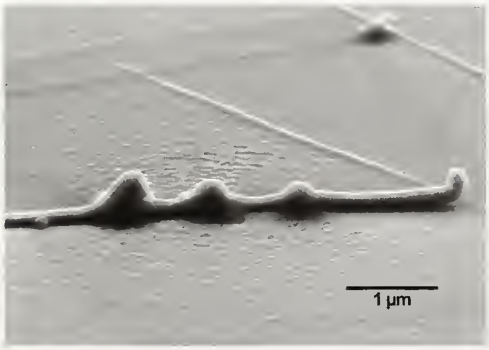

Figure 1: *SEM image of a 100 nm diameter multi-walled carbon nanotube attached by three welds to a Cr thin-film. The increase in size of the welds (from right to left) is due to increasing the time the electron beam was left focused on the nanotube.*

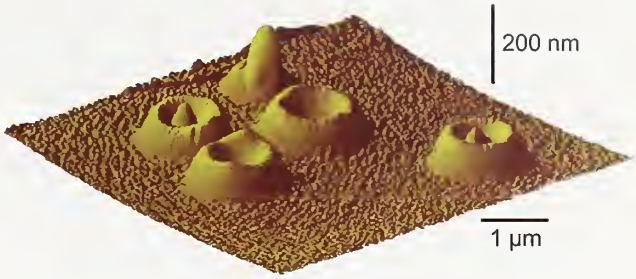

Figure 2: *AFM image of the effects of electron beam stigmation on EBD weld structures. The spike in the back is the result of a well-aligned electron beam.*

The quality of the EBD weld is highly dependent on the skills of the SEM operator. In FY03, we have been exploring the effects of beam collimation and stigmation on the weld. Figure 2 shows the variety of EBD weld structures that can be formed with different beam adjustments. Optimizing these variations in weld characteristics is an important step in ensuring the fabrication of high-performance interconnects.

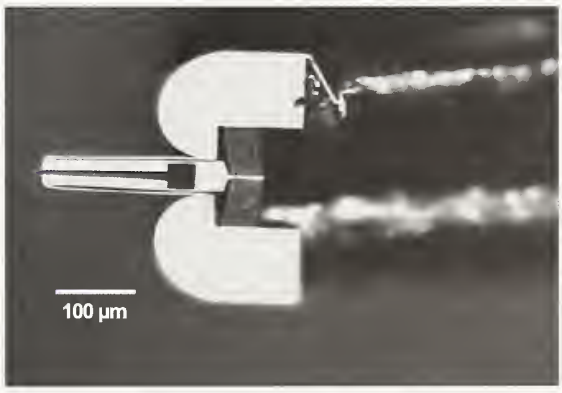

Figure 3: *Optical micrograph of tweezers for manipulating carbon nanotubes. The tips of the tweezers on the left are only a few microns long. They are actuated by thermal expansion of the silicon as an electrical current heats specific parts.*

In addition to controlling the interconnect fabrication process, manipulation of these small-scale structures is also critical for property characterization of these early-stage materials. With the help of researchers at the University of Colorado, we are developing tweezers capable of manipulating individual nanotubes for fabrication as well as characterization (see Figure 3).

Contributors and Collaborators

D.T. Read, R. Geiss, D.S. Finch (Materials Reliability Division, NIST); S.E. Russek, A.B. Kos (Magnetic Technology Division, NIST); A. Hartman (University of Colorado)

Physical Properties of Thin Films and Nanostructures: Carbon Nanotube Composites

Carbon nanotube reinforcements can dramatically improve a wide range of material systems. Even at low volumes, nanotube additions can significantly impact electrical conductivity, heat dissipation, and mechanical durability. A key component of the high performance of nanotube composites is the ability to effectively disperse and/or align individual nanotubes within the matrix. This project addresses new measurement approaches that can bring to light information on tube distribution and alignment and provide important data for process optimization.

Stephanie A. Hooker

Carbon nanotubes (CNTs) have received considerable attention recently due to their remarkable combination of mechanical, electrical, and thermal properties. One avenue to effectively utilize these unique materials is to combine them with polymers, thereby creating *nanocomposites*. Incorporation of even small volumes of CNTs into various polymers has already led to demonstrated increases in electrical conductivity, Young's modulus, and hardness as compared to the non-reinforced matrix. As a result of these early findings, applications in multifunctional composites, EM shielding, static dissipation, thermal isolation, and flaw detection are being pursued.

However, one major challenge associated with the development of CNT composites is effective distribution of individual tubes within the matrix. To investigate the effects of this distribution on composite performance, various fabrication techniques are being evaluated, and the resulting mechanical, thermal, and electrical properties are being correlated with microstructure features such as tube alignment and tube-to-tube contact. In addition, impedance analysis is being investigated as a potential method to obtain critical information related to the tube-matrix interface.

Three approaches to composite fabrication have been selected for evaluation in this project: mechanical mixing, doctor-blade tape casting, and templated synthesis from the gas phase. Based on initial discussions related to the different processes, composite specimens with tube distributions ranging from completely random to fully aligned should be feasible.

In FY03, specimens were prepared by mechanically mixing 0 to 1 weight % single-walled nanotubes (SWNTs) with a polymer commonly used in microelectronic packaging. Impedance characteristics were determined from 100 Hz to 1 MHz and used to calculate parameters such as electrical conductivity (Figure 1). For this specific process, the data indicated that the individual nanotubes are randomly distributed, and percolation has not yet been achieved. Process variations for improved nanotube incorporation are currently in progress.

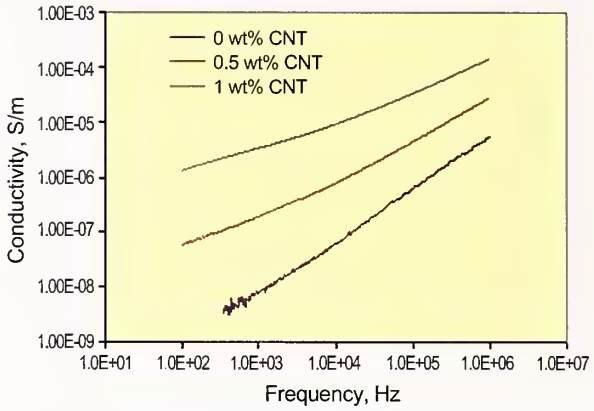

Figure 1: *Electrical conductivity of mechanically combined composites as a function of frequency. Specimens were provided by the University of Colorado at Boulder.*

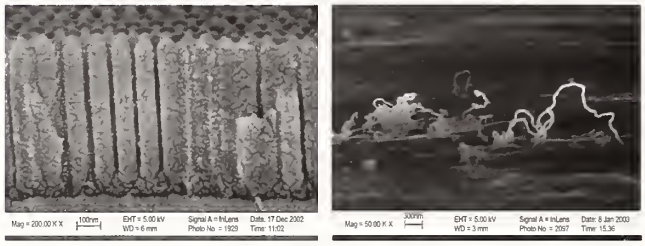

Figure 2: *Blank AAO template (left) and preliminary nanotubes grown inside and extending from an AAO template (right). Specimens were provided by Nanomaterials Research, LLC.*

In addition to randomly oriented composites, CNT arrays synthesized within the regularly aligned nano-sized pores of anodic aluminum oxide (AAO) (Figure 2) are also of interest for more advanced electrical characterization. Preliminary specimens have exhibited growth both within and beyond the porous template. Current research is focused on developing methods to contact and probe these CNT arrays.

Contributors and Collaborators

D. Finch, R. Geiss, P. Rice (Materials Reliability Division, NIST); D. Routkevitch, J. Alexander, O. Polyakov (Nanomaterials Research, LLC); V. Sundaram R.L. Mahajan (University of Colorado at Boulder)

Interface of Materials with Biology

New materials and devices are radically changing the medical treatment of injury and disease, yet because of the rapid pace of this segment of the materials industry, an adequate measurement infrastructure does not yet exist. The program on the Interface of Materials with Biology develops measurement methods, standards, and fundamental scientific understanding at the interface between materials science and biological science. Within the health care industry, we focus on dental and medical sectors that apply synthetic materials for replacement, restoration, and regeneration of damaged or diseased tissue. Three primary foci exist within this program: biocompatibility, materials properties, and materials science techniques applied to biological systems.

Whether the medical issue involves implanting a hip or knee joint prosthesis, a synthetic bone graft, or a tissue engineering scaffold into the human body, one of the primary issues is biocompatibility. By using our expertise in materials science, we are working to develop suitable Reference Materials (RM) for investigating biocompatibility and implant suitability. Research has focused on measuring the cellular response to powders and bulk materials to identify suitable candidates. We are collaborating with the American Dental Association Foundation (ADAF) to develop metrology methods to characterize the biocompatibility of synthetic bone grafts. Quantitative methods being developed include assays for adhesion, viability, proliferation, and differentiation of bone cells, as well as optical coherence tomography and confocal microscopy for measuring tissue ingrowth. We are developing biochemical assays to quantify inflammatory responses to synthetic materials. Finally, current research is working to bridge the gap between knowledge generation by cell biologists and product development in industry. In collaboration with the Chemical Science and Technology Laboratory, we are developing measurement methodologies and reference materials to use in assessing interactions in complex systems of living cells with synthetic materials. The expected outcomes of this work are reference substrates that induce specific cellular responses and engineered DNA vectors to act as fluorescent reporters of cellular responses.

In addition to the issue of biocompatibility, it is critical that the materials can withstand the mechanical and environmental stresses placed on them. For metallic implants, one concern is the corrosion pitting resistance of the implant materials and the associated potential for stress corrosion cracking (SCC). To address this issue, metal standards are being subjected to a simulated biological environment which will then be used to develop tests to assess the susceptibility for SCC.

Mechanical properties issues also arise when considering synthetic bone grafts and tissue engineering scaffolds. In addition to traditional bulk mechanical property measurements, combinatorial approaches are used to identify compositions and surface features that affect properties such as biocompatibility and mechanical durability. Finally, because the dental industry is primarily composed of small manufacturers with limited R&D capability, collaborations with the ADAF, located in MSEL, are filling the gap by developing improved materials and techniques, patenting and licensing these inventions, and, most importantly, providing a technical foundation. Research focuses on improved understanding of the synergistic interaction of the phases of polymer-based composites and the mechanisms of adhesion to dentin and enamel. This approach will ultimately lead to materials with improved durability, toughness, and adhesion to contiguous tooth structure.

In this era of interdisciplinary approach to research, we provide an added dimension. By taking a physical/mechanical approach to how cells function, respond, and remodel in interaction with synthetic materials, we can provide skill sets typically absent in the biomedical community. Our concentration on mechanical property metrology extends to biological systems, spanning a considerable size range from individual neurons and muscle cells to complete pulmonary arteries. This necessitates the development of unique mechanical testing platforms and application of a materials science approach to understanding integrated properties.

Fundamental to much of the work in this program is the recognition that surfaces and interfaces play a critical role in biological systems and, in particular, in the interactions of synthetic or designed materials with biological systems and function. By applying the expertise in the NIST Materials Science and Engineering Laboratory to characterization of surfaces and interactions at interfaces in biomaterials, we will accelerate the introduction of improved materials and help provide the means to assure quality control that is critical to this industry.

Contact: Timothy P. Quinn

Biomaterials Metrology: Pediatric Pulmonary Hypertension

Pulmonary hypertension is a potentially fatal complication of congenital heart defects in children who live at high altitudes. Discovering the cause and expression of the disease may open the way to finding new, more effective treatments. Our contribution to this goal is to provide data on the mechanical properties of the pulmonary artery and its constituents in healthy and diseased tissues.

Elizabeth Drexler, Christopher McCowan, Andrew Slifka, and Joyce Wright

Background

Children born with heart defects (approximately 1 %) at the high altitudes found in the Rocky Mountain region have an increased risk of developing pulmonary hypertension. The mechanisms for developing this potentially fatal complication are not clearly understood, nor are the physical modifications that result in clinical expression. It is known that with the hardening of the pulmonary artery (PA), the heart becomes enlarged trying to maintain a constant flow volume. This, in turn, exacerbates the condition of the artery and eventually leads to congestive heart failure.

Technical Strategy

The overall goal of the project is to determine the critical factors in the development of pulmonary hypertension in children so that we can learn how to prevent or, at least, mitigate the effects. Toward that end, it is necessary to characterize the fluid dynamics of the system and identify how, where, why, and over what time period the tissue of the artery remodels. The "how" will be answered through measurement of the mechanical properties and how they change with the onset of hypertension; the "where" through the histology of the artery; the "why" through study of the biochemical, proteomic, and genomic signals that cause the tissue to remodel; and the time frame by using input from all the preceding. Our contribution will be to measure and compare the mechanical properties and histology of PAs using animal models. Our proximity to the sources for the tissue allows us to test within 24 hours of excising the arteries, so the cells are still viable and responsive. We have chosen a bubble test technique that can scale easily to accommodate the different geometries that we will be testing. It also permits us to keep the tissue nourished and hydrated without compromising the quality of the data. A computer controlled test system provides excellent control and rapid data acquisition. The most recent test system is shown in Figure 1.

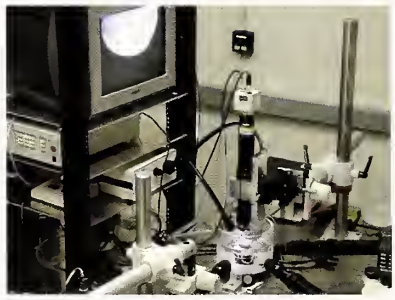

Figure 1: *The computer-controlled, automated test system for measuring the biaxial mechanical properties of normo- and hypertensive pulmonary arteries.*

Accomplishments

We began with testing the main PA of Sprague–Dawley rats, both controls and those injected with monocrotaline to induce hypertension. By the end of March, we had received 24 specimens and successfully completed testing of 7 controls and 9 monocrotaline-affected specimens up to pressures of 17.9 kPa. Usefulness of the data was limited due to the disparity of our test population. Upon consulting with our collaborators, it was decided to continue the test program with Long–Evans male rats, approximately 12 weeks old, and to compare the properties of the main and proximal PAs. A third group was added to the test suite: genetically altered hypoxic Long–Evans rats that lack the endothelium B marker, which display a propensity for developing pulmonary hypertension. We have completed testing of the controls and monocrotaline PAs, and the preliminary analysis indicates that there is a difference in the stress–strain behavior that shows more compliance in the control arteries, particularly in the longitudinal direction.

In addition to the tests on the rat model, we have completed some initial tests using a calf model. We have tested 2 normotensive calf PAs, main and proximal, using a larger version (25 mm diameter aperture) of our bubble test fixture. We will soon begin testing the PAs from hypoxic calves to quantify their mechanical properties.

Contributors and Collaborators

B.J. Filla, D.S. Finch, T. Oreskovic, L. Rodine (Materials Reliability Division, NIST); R. Shandas, M. Salehi (University of Colorado); K. Colvin, D. Ivy (University of Colorado Health Sciences Center and Children's Hospital of Denver)

Biomaterials Metrology: Mechanical Response of Tissue Engineering Constructs

Tissue engineering offers the hope that diseased or injured structures within the body can be replaced by tissues grown on scaffolds. To be effective, test methods for the mechanical properties of the polymeric scaffold, with and without tissue ingrowth, must be developed and the properties themselves measured.

Timothy P. Quinn and Tammy L. Oreskovic

Background

A proposed method for treating disease and injury in the human body is to replace the diseased tissue with tissue that is grown outside the body. Typically, a seed of healthy tissue is cultured to grow into a polymeric or natural scaffold. The scaffold supports the tissue and coaxes it to grow into the proper geometrical shape. The tissue and scaffold are then implanted in the patient to replace the injured or diseased tissue.

It is essential to know the mechanical response of the scaffold before, during, and after tissue is grown in it. Tissue engineered (TE) bone replacements must have the proper stiffness and strength to carry the structural loads applied to the body. Vascular grafts must support the pressures applied to the cardiovascular system. It has been discovered that both of the TE constructs mentioned above must be grown under a state of mechanical stress, or else the resulting properties of the construct do not approach those of natural tissue.

Mechanical Measurements

The Polymers Division provided us with poly(e-caprolactone) (PCL), a biodegradable polyester, that had been co-extruded with poly(ethylene oxide) (PEO). We molded 3 mm cubic samples and cured them for various amounts of time. The water soluble PEO was then washed-out of the samples. The time that the samples were cured controlled pore size and the pore size controlled the mechanical properties.

A servo-hydraulic, materials-testing machine was outfitted for compression testing of TE scaffolds. The displacement of the hydraulic actuator was measured with an LVDT. A video microscope imaged one face of the specimen during the test at about 75x. The samples were tested in displacement control with the crosshead velocity set to 3×10^{-3} mm/s for a nominal strain rate of 1×10^{-3}/s. The video images were captured every 2.14 s. Strain was calculated by image correlation and also using the output of the LVDT (as is done in many papers in this field) (Figure 1). A hyperbolic model represents the data well and has been used to compare scaffolds of various pore configurations (Figure 2).

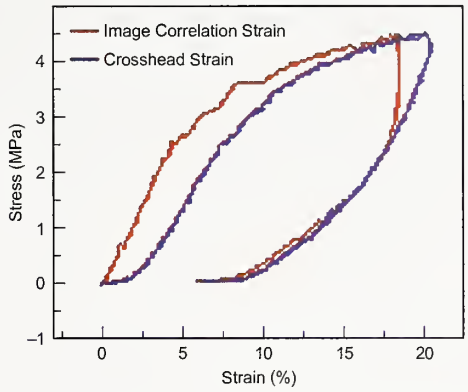

Figure 1: *Comparison of strain measured from image correlation and crosshead displacement. Note that measuring strain from crosshead displacement can lead to errors of up to 10 % of the strain.*

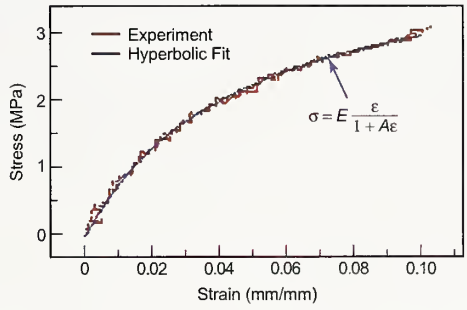

Figure 2: *The compression data were fit with a hyperbolic model.*

A Standard Test Frame

A standard test frame has been designed for tissue engineered vascular grafts that aims to isolate all of the independent variables which include local stress state and pore size, cyclic stress frequency, cyclic stress amplitude (with and without spikes), duration of cyclic loading, stress waveform (pulsatile *vs.* cyclic effects), scaffold impregnation (collagen, laminin, etc.), culture medium flow conditions, passage, biochemical supplements, and cell source.

Contributors and Collaborators

J.D. McColskey, C.N. McCowan (Materials Reliability Division, NIST); N.R. Washburn (Polymers Division, NIST); C.D. Cool (University of Colorado Health Sciences Center)

Biomaterials Metrology: Cellular Engineering Microsystems (CEMS)

The biomedical community, both academic and commercial, seeks a research platform for the study of biochemical and physical processes at the single cell scale for understanding cellular response to a range of stimuli (chemical, mechanical, optical, electromagnetic). We are in the process of developing CEMS to address this perceived need to enable the stimulation of single cells and cellular arrays including the direct measurement of cellular response such as changes in modulus.

Dudley S. Finch

Background

The objective of Cellular Engineering Microsystems (CEMS) is to utilize MEMS-based technology to enable stimulation (chemical, electrical, optical, mechanical, etc.) of single cells and arrays as well as a means of sensing their response. For example, chemical stimulation of leukocytes results in changes in the mechanical properties of the cell. The leukocyte is spherical and must undergo large deformations as it flows through narrow pulmonary capillaries. Any change in mechanical properties has a direct impact on the number of cells available in the lung tissue and hence affects the immune response. The study of changes in mechanical properties with different chemical stimuli provides valuable information on the factors affecting cell motility and, hence, transit times in the microvascular system.

Accomplishments

A number of key accomplishments have been made in the last year. The early designs of CEMS have been tested and revised. The current generation of CEMS for the measurement of the contractile forces of vascular smooth muscle cells (VSMC's) is shown in Figure 1. Testing of the early prototypes revealed problems with sensitivity as well as reliability of the devices leading to the device shown. In this figure, only one pad is used for cellular adhesion with sensing and actuation being achieved via a dual comb drive approach. The pad in this case is split into four pieces. The gap between the pieces (around 2 micrometers) can be alleviated by applying a bias to the comb drives thereby moving each of the wedges 1 micrometer to close the cap. This design is limited to a total displacement of approximately 5 micrometers, allowing up to 10 % strain to be applied on a typical VSMC. To date, VSMC's have been shown to adhere and spread on the pads which were coated with atomic layer deposited (ALD) alumina. We are in the process of calibrating the devices and integrating them with adhered VSMCs. Still to be tested, however, is their functionality and sensitivity in a buffered fluidic environment.

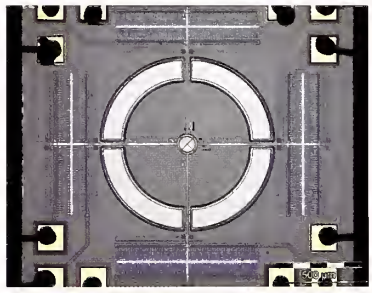

Figure 1: *A packaged CEMS device incorporating comb drives for the measurement of VSMC contractile forces.*

Another key development is the application of CEMS to the measurement of cellular adhesion. Figure 2 shows a "flip chip" cell pusher adjacent to a melanoma cell. This device is based on a thermal actuator and provides nanonewton forces with displacements up to 30 micrometers. This CEMS device is being extended to include the direct probing of cells in order to measure mechanical properties of leukocytes.

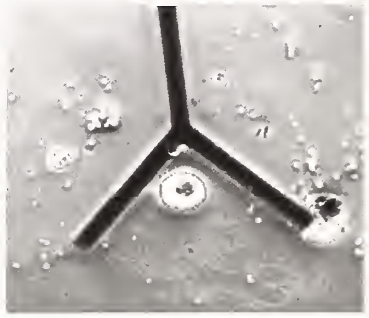

Figure 2: *A flip chip thermally actuated cell poker shown adjacent to a melanoma cell.*

Calibration of the forces generated via the CEMS devices is currently being developed with the use of an optical tweezer with a force control loop.

Contributors and Collaborators

A. Slifka, C. McCowan, D. Serrell, C. Bender (Materials Reliability Division, NIST); P. Jones, R. Nemenoff, K. Pfenninger (University of Colorado Health Sciences); G.S. Worthen (National Jewish Medical and Research Center); D. Marr (Colorado School of Mines)

Biomaterials Metrology: Cellular Level Measurements

Techniques and tools that facilitate the exposure of single cells (and arrays) to controlled and quantifiable mechanical forces, and, at the same time, allow for the characterization of other biological phenomena, are needed for the study of cardiovascular tissues and systems. The development and evaluation of one of these tools, an optical tweezer, is a focus of this year's effort.

Christopher McCowan and Andrew Slifka

Overall, the challenge is to develop mechanical test platforms and tools that can be integrated with currently used biological techniques for the evaluation and measurement of cellular response (*e.g.*, gene expression, cell morphology, area of adhesion, etc.). These types of studies are needed because the development of vascular smooth muscle cells in cardiovascular tissue, for example, is dependent on the variations in the stress–strain environment that result from the expansion and contraction of the vessel wall. The importance of the environment becomes apparent when one considers that engineered tissues, so far, have mechanical properties that differ from naturally grown tissues. This is possibly a bulk effect but is clearly related to processes at the cellular level. Without a quantitative understanding of the mechanics and functionality of the building blocks (cells and fibers), the bulk properties of the tissues cannot be fully understood and modeled.

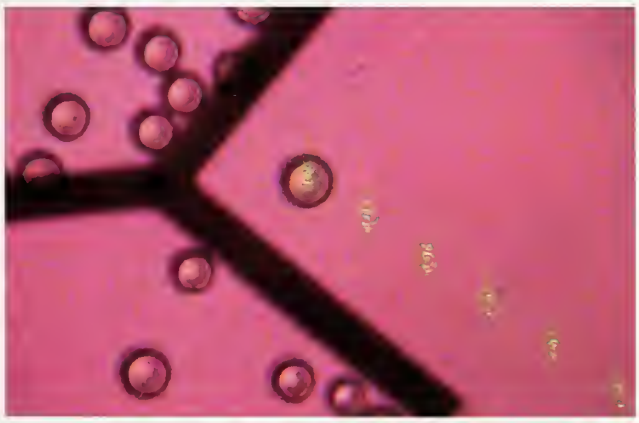

Figure 1: *A polystyrene ball (15 mm) in the optical trap being positioned to push on the y-shaped actuator of the bio-MEMS device.*

The focus for this year was to design, build, and calibrate a force/displacement measurement loop onto an existing optical tweezer, and use this tweezer to calibrate a MEMS device designed for testing single cells. To facilitate this effort, we are collaborating with David Marr at CSM, who has two optical tweezers. To date, we have written and tested the control code for our system and can accurately position the optical trap. Currently, code is being developed to acquire and monitor the position of the bead within the trap (force feed-back loop), and a fluid flow channel for the calibration of the tweezer is being built. This flow channel will allow us to calibrate the tweezer using a drag force or Stokes method and will be compared with Brownian motion-based methods of calibration.

The force of the optical trap is expected to be in the nano- to pico-newton range. We had initially planned to use the tweezer to calibrate a MEMS device designed for cellular testing (Figure 1). The first generation devices, which push the limits of MUMPS technology, will likely be too stiff for direct calibration with the tweezer. Therefore, we are pursuing alternate calibration methods for these initial MEMS devices (100's of nN force range).

Our first application of the tools developed in this program will be to measure mechanical properties of leukocytes. This will be done in collaboration with Dr. S. Worthen of National Jewish Medical and Research Center, who has been studying the role of polymorphonuclear leukocytes, or neutrophils, in acute lung inflammation. The neutrophil is larger than the capillaries in the vascular bed of the lung. Therefore, details of the deformation of the neutrophil and capillaries are important in determining the mechanisms of pulmonary inflammatory response. We plan to develop tools and MEMS devices to apply and sense deforming forces on cells such as neutrophils.

Contributors and Collaborators

D. Finch, D. Lauria (Materials Reliability Division, NIST); R. Rorrer (Mechanical Engineering, CU Denver); D. Marr, J. Oakey (Chemical Engineering, Colorado School of Mines); D. Serrell, C. Bender (Mechanical Engineering, CU Boulder)

Materials for Micro- and Opto-Electronics

U.S. microelectronics and related industries are in fierce international competition to design and produce smaller, lighter, faster, more functional, and more reliable electronics products more quickly and economically than ever before. At the same time, there has been a revolution in recent years in new materials used in all aspects of microelectronics fabrication.

Since 1994, the NIST Materials Science and Engineering Laboratory (MSEL) has worked closely with the U.S. semiconductor, component, packaging, and assembly industries. These efforts led to the development of an interdivisional MSEL program committed to addressing industry's most pressing materials measurement and standards issues central to the development and utilization of advanced materials and material processes. The vision that accompanies this program — to be the key resource within the Federal Government for materials metrology development for commercial microelectronics manufacturing — may be realized through the following objectives:

- Develop and deliver standard measurements and data;
- Develop and apply *in situ* measurements on materials and material assemblies having micrometer- and submicrometer-scale dimensions;
- Quantify and document the divergence of material properties from their bulk values as dimensions are reduced and interfaces contribute strongly to properties;
- Develop models of small, complex structures to substitute for or provide guidance for experimental measurement techniques; and
- Develop fundamental understanding of materials needed in future micro- and opto-electronics.

With these objectives in mind, the program presently consists of projects led by the Metallurgy, Polymers, Materials Reliability, and Ceramics Divisions that examine and inform industry on key materials-related issues. These projects are conducted in concert with partners from industrial consortia, individual companies, academia, and other government agencies. The program is strongly coupled with other microelectronics programs within government and industry, including the National Semiconductor Metrology Program (NSMP) at NIST. Materials metrology needs are also identified through industry groups and roadmaps, including the International Technology Roadmap for Semiconductors (ITRS), the IPC Lead-free Solder Roadmap, the National Electronics Manufacturing Initiative (NEMI) Roadmap, the Optoelectronics Industry Development Association (OIDA) roadmaps, and the National [Magnetic Data] Storage Industry Consortium (NSIC).

Although there is increasing integration within various branches of microelectronics and optoelectronics, the field can be considered in three main areas. The first, microelectronics, includes needs ranging from integrated circuit fabrication to component packaging to final assembly. MSEL programs address materials metrology needs in each of these areas, including lithographic polymers and electrodeposition of interconnects, electrical, mechanical, and physical property measurement of dielectrics (interlevel, packaging, and wireless applications), and packaging and assembly processes (lead-free solders, solder interconnect design, thermal stress analysis, and co-fired ceramics).

The second major area is optoelectronics, which includes work which often crosses over into electronic and wireless applications. Projects currently address residual stress measurement in optoelectronic films, optical and structural characterization of wide bandgap semiconductors, and standards development for III–V compound semiconductors. Cross-laboratory collaborations with EEEL figure prominently in this work.

The third area is magnetic data storage, where the market potential is vast and growing and the technical challenges extreme. NSIC plans to demonstrate a recording density of 1 terabit per square inch — 40 times today's level — by 2006. To reach these goals, new materials are needed that have smaller grain structures, can be produced as thin films, and can be deposited uniformly and economically. New lubricants are needed to prevent wear as spacing between the disk and head becomes smaller than the mean free path of air molecules. Some measurements require calibration of magnetometers using certified magnetic standards in several different shapes and magnetic strengths, and with a wide range in magnetic character. MSEL is working with the magnetic recording industry to develop measurement tools, modeling software, and standards to help achieve these goals, with MSEL, the Electronics and Electrical Engineering Laboratory, the Physics Laboratory, the Information Technology Laboratory, and the Manufacturing Engineering Laboratory working as partners in this effort.

Contact: Robert R. Keller

Electronic Packaging and Components: Packaging Reliability

We are developing methods to examine materials and interfaces in electronic packaging applications and elucidate the damage mechanisms. Our current focus is on advanced packaging structure, embedded passive materials, and thin metal films using thermal microscopy to measure heat flow and thermal properties on increasingly smaller size scales.

Andrew Slifka

Technical Description

The microelectronics industry is moving toward higher density components of smaller size using less expensive materials. One move in this direction is the advent of embedded and integrated passive components in printed circuit boards (PCBs). These organic-based PCBs can have a large coefficient of thermal expansion (CTE) in comparison with many materials found in proximity. This CTE mismatch can reduce the reliability of electronic packaging systems by causing localized stress.

In addition, thermal issues are becoming increasingly important as size decreases and functionality increases. Applications in the microelectronics industry would include heat removal and interconnects.

We are investigating the damage induced from CTE mismatches between organic materials, organics and metals, and organics and ceramics to determine the initiation of damage and the ultimate failure mechanisms in these systems. Thermal microscopy is used to measure changes in interfacial thermal resistance in order to detect the onset of thermomechanical damage at that interface before any surface manifestation is visible.

We are developing new measurement methods using scanned-probe microscopy (SPM) in order to characterize packages and measure thermal conductivity of thin films at increasingly smaller size scales.

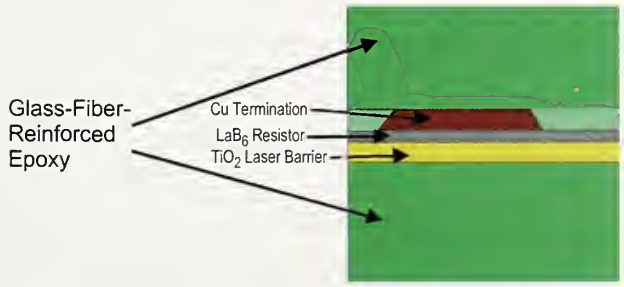

Figure 1: *Schematic of an industrial embedded resistor sample.*

Accomplishments

We are continuing measurements on industrial embedded resistors (Figure 1), making comparative measurements between the SPM thermal system and the IR microscope. We have purchased a new IR microscope that has better accuracy than our old system, but the measurement protocol has remained the same.

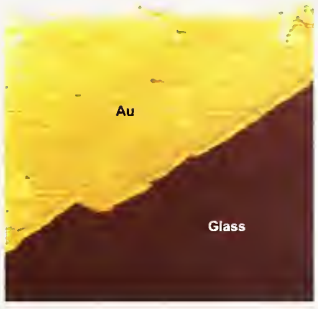

Figure 2: *Thermal SPM image of a 150 nm gold film on glass.*

We have begun measurements of films using thermal SPM in a mode that uses the probe tip as a point-source heater, which is kept at a constant temperature because the probe tip is a resistive element in a Wheatstone bridge circuit. Heat from the probe tip is conducted into the sample, and as a film becomes thinner, heat loss from the probe tip is due more to the substrate rather than to the film. Dr. K. Cole has developed a theory that accounts for this film effect. We have been making preliminary measurements using various thicknesses of gold films on different substrates and comparing the theory to our measurements. Figure 2 shows a 150 nm thick gold film on a glass substrate. We will add interfacial modifications to the samples and to the theory in order to make the measurement method applicable to films used in industry. In addition, we have been using this measurement method on diamond-like-carbon (DLC) films. These industrial films are made from a polymer precursor, which allows easy and inexpensive coating onto various substrates. The thermal conductivities of these films, and how they compare to conventionally processed DLC films, is being investigated.

Contributors and Collaborators

John Felten (DuPont Technologies Research, Triangle Park, NC); Richard Snogren (SAS Circuits, Inc., Littleton, CO); Dr. Kevin Cole (University of Nebraska); Charles Partee (Cenymer Corporation, Longmont, CO)

Electronic Packaging and Components: Acoustic Loss in Piezoelectric Crystals

Langasite, langatate, and langanite are piezoelectric materials that show promise of providing characteristics superior to quartz for a variety of electronic oscillator and filter applications. The acoustic loss of these crystals is being studied in our laboratory to identify and quantify the dominant physical mechanisms that degrade performance. Through this work, the project seeks to provide guidance to other researchers in minimizing the acoustic loss and determining the most promising material to pursue among this group of compounds.

Ward Johnson and Sudook Kim

Technical Description

Langasite ($La_3Ga_5SiO_{14}$) and its isomorphs, such as langatate ($La_3Ga_{5.5}Ta_{0.5}O_{14}$) and langanite ($La_3Ga_{5.5}Nb_{0.5}O_{14}$), have attracted significant attention in recent years as materials for improved electronic oscillators and filters. The advantages of these crystals over quartz include lower acceleration sensitivity; higher piezoelectric coupling, which enables devices to be made smaller; and higher Q, which reduces phase noise and enables higher-frequency operation. These materials also have no phase transition below the melting point, which allows devices to be operated at high temperatures and provides the potential of producing large-diameter wafers (for surface-acoustic-wave devices) more easily and cheaply than with quartz.

The acoustic loss Q^{-1} in langasite, langatate, and langanite at ambient temperatures has been found to vary significantly between specimens, even when taken from the same crystal boule. This research project has provided information on the physical sources of these variations in each of these materials through measurements of Q^{-1} as a function of temperature T and frequency.

Accomplishments

Measurements of the acoustic loss of trapped resonant modes of Y-cut plano-convex disks of langasite, langatate, and langanite were performed between 100 K to 740 K. Figure 1 shows an example of measurements on langasite with the minimum value of measured Q^{-1} (Q^{-1}_{min}) subtracted. Q^{-1}_{min} occurs near the lowest measured temperature, 100 K, and is ~1 x 10^{-6} at each frequency. Anelastic relaxation peaks appear on top of a background that increases with an approximate Arrhenius temperature dependence. These features are similar to those observed in langatate and langanite.

Activation energies determined from the peak positions of the lowest-temperature relaxations in langasite and langatate (assuming a Debye form) are 0.34 ± 0.05 eV and 0.25 ± 0.03 eV, respectively, which are typical of point-defect relaxations. The lower value of the activation energy in langatate causes the peak to appear at lower temperatures. This difference may provide an explanation for results in the literature that show the room-temperature Q to be generally higher in langatate than in similarly grown and fabricated langasite.

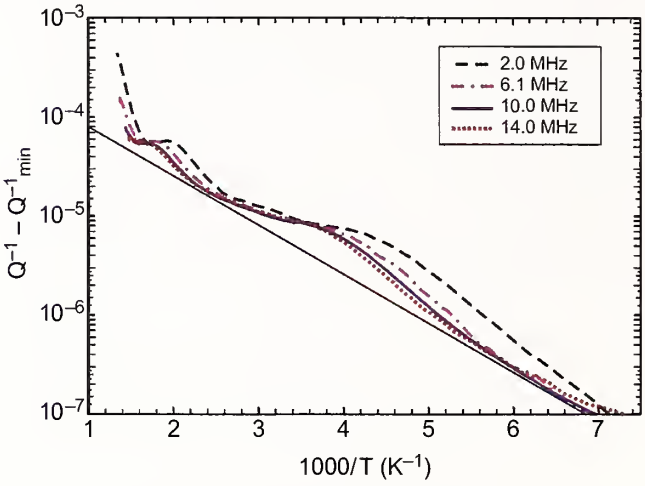

Figure 1: *Q^{-1} versus 1000/T of a langasite specimen with the minimum Q^{-1} at each frequency (Q^{-1}_{min}) subtracted.*

The ultimate limit of the Q in piezoelectric crystals is determined by phonon–phonon interactions (Akhieser mechanism), which produce a loss that is proportional to frequency and weakly dependent on temperature above ambient temperatures. The results shown in Figure 1 and similar results for langatate and langanite show no evidence for such a contribution to Q^{-1}. The lack of an obvious intrinsic contribution suggests that the maximum attainable Q of these materials may be significantly higher than values reported thus far in the literature.

Contributors and Collaborators

Robert Smythe (Piezotechnology, Inc.); Satoshi Uda (Tohoku University)

(This project was partially supported by a grant from the U.S. Army Research Laboratory.)

Micrometer-Scale Reliability: Mechanical Behavior of Thin Films

Measurement methods and materials data are needed for the design of interconnect structures in high-performance integrated circuits. These micro- and nanometer-scale thin films are formed by physical vapor deposition; their microstructures, and hence their mechanical properties, are quite different from those of bulk materials of the same chemical composition. While the general principles of conventional mechanical testing are applicable to thin films, special test equipment and techniques are required. The ultimate goal of this project is to characterize the exact materials used in IC fabrication, at their proper size scale.

David T. Read

Interconnect structures in advanced integrated circuits carry power, signals, and heat from the transistors to the outside environment. These structures consist of multiple layers of thin films of conductors and dielectrics with barrier and adhesive layers. These thin films are an essential component of all advanced electronic devices, and similar materials are used in a variety of other applications, such as reflective coatings. Industry is aggressively pressing new materials into service, reducing the size scale of the structures, and requiring more functionality, including mechanical functionality, from all components of their structures. Design of reliable structures relies on quantitative numerical modeling, which requires accurate material property data. Because the films are formed by physical vapor deposition, their microstructures, and hence their mechanical properties, are quite different from those of bulk materials of the same chemical composition. Techniques for measuring the mechanical behavior of thin films are being developed and applied.

The objectives of this project are:

- To develop experimental techniques to measure the mechanical properties of thin films, in specimens fabricated and sized like materials used in actual commercial devices;
- To relate the mechanical behavior of thin films to microstructure;
- To extend test techniques from their present level (1 μm thick, 10 μm wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths of 0.1 to 1 μm).

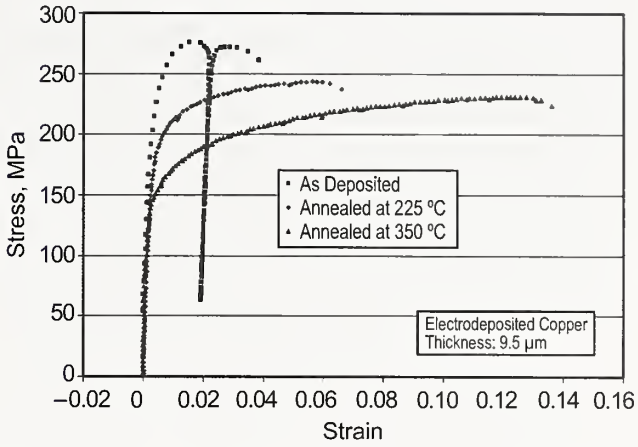

Figure 1: *Stress–strain curves for thick electrodeposited copper.*

In previous years, we reported properties of aluminum, polyimide, and polysilicon films at room temperature, including the special techniques developed to produce and test the specimens. This year, we obtained copper films from an industry collaborator and also developed procedures to electrodeposit copper films in-house for comparison. The thicknesses ranged from 1 to 10 micrometers thick. Photolithography of copper is a challenge which industry has worked around by implementing the damascene process. We used photolithography to make specimens up to 20 micrometers wide from the thicker copper electrodeposits, and our standard 10-micrometer-wide specimens from the thinner films, but making testable specimens was a big part of the effort of this project.

As part of this collaboration, nanoindentation data were also obtained on one series of films. While it is clear that nanoindentation can characterize the general mechanical robustness of a material, the quantitative correlation between nanoindentation data and microtensile data has not been established. In this study, our collaborators performed nanoindentation tests and applied the relatively new procedure of measuring the size of the plastic zone around the indentation with an atomic force microscope (AFM). The yield strengths that they obtained matched up well with the microtensile data shown here.

Contributors and Collaborators

Y.-W. Cheng, J.D. McColskey, R.R. Keller, R. Geiss (Materials Reliability Division, NIST); R. Emery, F. Hua, T. Scherban (Intel)

Micrometer-Scale Reliability: Stress Voiding and Electromigration

Reliability of the small-scale materials that make up advanced chip-level interconnect systems is not readily predictable by extrapolating known behavior for larger scale materials. We concentrate on studies of the failure mechanisms associated with thermal and electrical stressing, which limit performance in a variety of structures with constrained dimensions. Emphasis is placed on the roles of localized stress and variations in microstructure. We perform basic studies on aluminum-based interconnects, as well as measurements on advanced sub-100 nm systems comprising copper or silver.

Robert R. Keller and Roy H. Geiss

Stress voiding (SV) and electromigration (EM) remain as two primary reliability-limiting phenomena in chip-level interconnects, especially with the largely unexplored behaviors of copper/low-k systems. Both phenomena depend sensitively on the mechanical behavior of the metal and dielectric. Failures take the form of surface topography development and void formation, and can lead to open or short circuits. Our earlier work demonstrated that even small variations in localized microstructure can have large impacts on these types of failure.

This year, we have made significant progress in documenting the damage processes leading to failure from low-frequency, high-current-density AC stressing. The tests simulate the type of thermomechanical fatigue that can occur during a device's operational lifetime, due for instance to power cycling, energy saving schemes, and to application-specific thermal fluctuations. Fatigue, in this case, takes place because of cyclic Joule heating in the metal and because of a mismatch between thermal expansion coefficients of the metal and the substrate and/or passivation. Characterization, at present, primarily takes the form of orientation mapping by electron backscatter diffraction (EBSD).

Quasi *in situ* testing of an Al-1Si line revealed an evolution of grain size and orientation during 100 Hz testing at 12 MA/cm^2, as shown in Figure 1. The sequence shows the evolution of surface roughness, grain size, and crystal orientation during five minutes of stressing. Notable is the tendency for surface slip band offsets to form in larger grains. Their severity increased in grains that showed significant growth, such as the one on the left end of the EBSD maps. Other notable observations indicated that many grains changed their orientations during stressing, primarily those that also exhibited growth. This is seen in Figure 1 by the color maps on the right, indicating changes in transverse orientation; we note that virtually every grain maintained a surface normal of (111) throughout testing. The transverse orientation changes, however, led to an increasing number of grains with higher resolved shear stresses, assuming primary loading longitudinally. Such orientation changes facilitate roughness formation by allowing more dislocation slip activity. This particular line failed in 340 seconds, with an open circuit forming several micrometers away from the region shown above.

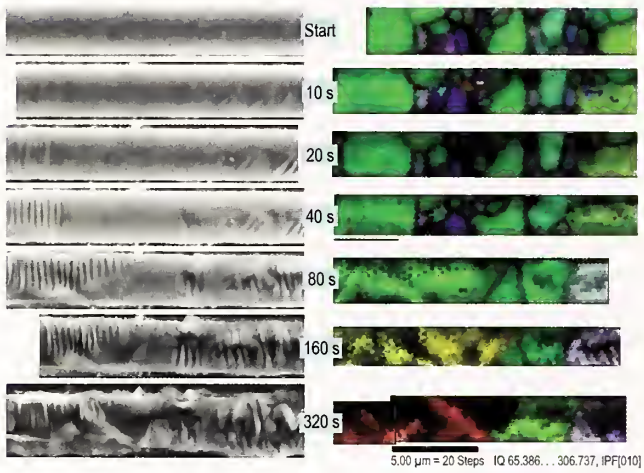

Figure 1: *SEM images and EBSD maps showing damage evolution during five minutes of severe AC stressing in Al-1Si.*

Our work on this long-term project appears to have significance in the three- to five-year timeframe for other researchers, as evidenced by a jump in non-self-citations in the peer-reviewed literature. As an example, for four papers published between 1996 and 1999, we were cited just two times within two years of publishing, but 22 times in 2001 and 2002.

Ongoing work includes more detailed TEM-based studies of the AC-induced damage. We are also measuring electrical and electromigration properties of extremely narrow lines of copper and silver fabricated by novel electrodeposition methods.

Contributors and Collaborators

Y.-W. Cheng (Materials Reliability Division, NIST); D. Josell, T. Moffat (Metallurgy Division, NIST); C. Witt (SEMATECH); R. Mönig, C. Volkert (Max–Planck–Institut für Metallforschung)

Micrometer-Scale Reliability: Strain in Photonic Semiconductors

The compound semiconductor photonics industry seeks to both measure and control strain that develops during the course of device fabrication. In the Materials Reliability Division, we are developing methods for measuring and mapping elastic strain distributions using electron diffraction. The methods are applied to phase transition-induced strains for the case of oxide formation in vertical cavity surface-emitting lasers, and to lattice parameter mismatch strains for self-assembled quantum dots.

Robert R. Keller and Roy H. Geiss

Vertical cavity surface-emitting lasers (VCSELs) present an attractive solution to many limitations faced by conventional edge-emitting lasers. Fabrication of aluminum oxide apertures for both electron and photon confinement in VCSELs is accomplished by wet thermal oxidation of AlGaAs between GaAs layers. Accompanying the transition is a volume contraction that exceeds 6 %. Thermal cycling during subsequent processing steps often results in delamination between the oxide and semiconductor interfaces, causing device failure. Our work with the Optoelectronics Division addresses the measurement and control of strain in VCSEL structures to minimize failure. We use both TEM and SEM-based electron diffraction to determine the strain states by means of measuring lattice spacings and angles. Compositional control during fabrication is used to tailor strain distributions. We describe here recent results on elastic strain mapping in the SEM.

Last year, we reported the exciting observation that electron backscatter diffraction (EBSD) pattern sharpness maps revealed a distorted region about the oxide growth front. Since that time, we have modeled the strain field using finite-element methods and have demonstrated mapping at much higher spatial resolution. Pattern sharpness shows a dependence upon all strain components since many reflections are used in the quantification. The shape and spatial extent of the distorted region in the resulting maps are consistent with the von Mises equivalent strain field expected about the oxide front, as determined by finite-element analysis. We are using kinematical electron diffraction theory to extract quantitative information regarding the magnitude of the strain gradient measurable in the patterns and maps.

Figure 1 shows an example of a high-resolution map where the beam was stepped in 10 nm increments. It reveals in detail the gradient in pattern sharpness normal to the interfaces (redder pixels indicate sharper patterns). We have determined that the orientation-dependent information volume for backscattered electrons has a diameter of approximately 25–30 nm in GaAs for the 15 kV electrons used in this scan. This map is therefore approaching the limit in terms of spatial resolution for this strain mapping method. We note that the resolution limit is somewhat smaller than the information volume diameter since we can overlap the sampling volume by means of the small step size and still detect differences from point to point. We are exploring improved resolution by decreasing the electron energies and by experimenting with camera positions.

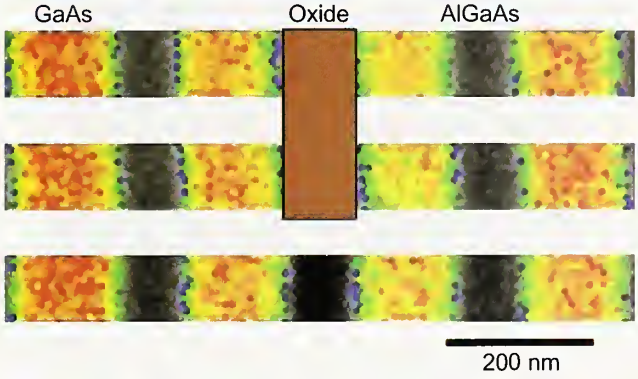

Figure 1: *High resolution EBSD pattern sharpness map, with 10 nm step size.*

This year, we began presenting some of our metrology results at professional society-sponsored conferences including the TMS Annual Meeting, the Summer Meeting of the Applied Mechanics and Materials Division of ASME, and the TMS Electronic Materials Conference. The prospect for using EBSD to both measure and map elastic strain distributions generated considerable interest, especially among those familiar with the technique. We feel that these developments have potential application to areas outside the photonics industry as well.

Contributors and Collaborators

T. Quinn, V. Tewary (Materials Reliability Division, NIST); A. Roshko, S. Lehman, K. Bertness (Optoelectronics Division, EEEL, NIST)

Micrometer-Scale Reliability: Bridging Length Scales

We have developed a multiscale model for nanostructures in solids. The model relates the physical processes at the interatomic level to measurable lattice distortions at the nanometer level and macroscopic stresses and strains. The model links the subnano (interatomic), nano (nanostructures), and macro length scales by integrating the powerful techniques of molecular dynamics, lattice-statics Green's function, and continuum Green's functions.

David Read and Vinod Tewary

Technical Description

Mathematical modeling is a very important tool for understanding the mechanical behavior of nanomaterials and for research and design of devices based upon nanostructures. A nanostructure needs to be modeled at the following scales: (i) the core region of the nanostructure (sub nanometer) where the nonlinear effects may be significant; (ii) the region of the host solid around the nanostructure (nanometer); and (iii) free surfaces and interfaces in the host solid (macro). A nanostructure causes lattice distortion in the host solid that manifests as strain throughout the solid. The strain is essentially a continuum-model parameter whereas the lattice distortions are discrete variables that must be calculated by using a discrete lattice theory. Hence, one needs a multiscale model that relates the discrete lattice distortions at the microscopic scale to a measurable macroscopic parameter such as strain.

Conventional models of nanostructures are based upon either the continuum theory, which is not valid close to the defect, or molecular dynamics (MD) that is CPU intensive and usually limited to small crystallites, which may introduce spurious size effects. We need a computationally efficient multiscale model that links the length scales from subnano to macro and can be used on an ordinary desktop. Such a model will be a valuable tool for research and engineering designs.

Our model is based upon the lattice-statics Green's function (LSGF) that reduces asymptotically to the continuum Green's function (CGF). However, the response of the solid containing a nanostructure is given by the defect GF that does not reduce to the CGF. The defect GF ($\mathbf{G}^*$) is related to the perfect LSGF ($\mathbf{G}$) through the Dyson integral equation:

$$\mathbf{G}^* = \mathbf{G} + \mathbf{G}\Delta\Phi\mathbf{G}^*.$$

Using the Dyson equation, we can write the response of the defect lattice as a product of an effective Kanzaki force and $\mathbf{G}$,which links the perfect LSGF and the CGF.

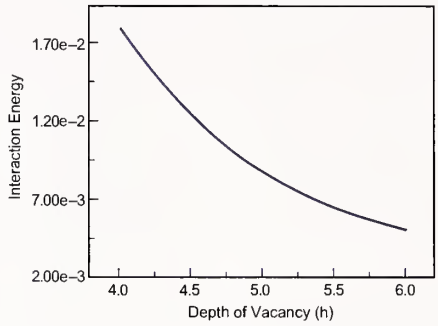

Figure 1: *Interaction energy (eV) between a vacancy and the free surface in Cu (h in units of a=1.807 Å).*

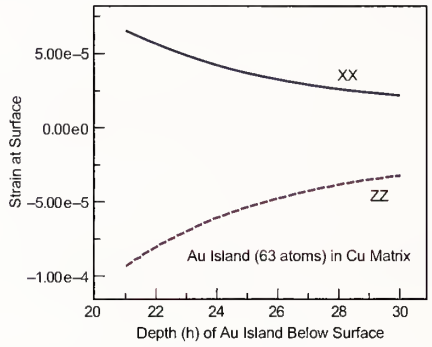

Figure 2: *Strains at the free surface due to a gold nanoisland in Cu.*

Accomplishments

We have developed a multiscale Green's-function (MSGF) method that integrates LSGF and CGF and applied it to model point defects in metals and semiconductors. This method is suitable for a weak defect such as a vacancy. For modeling nanostructures and other strong defects, we have integrated this method with modified MD in the core of the nanostructure. This integration makes our model truly multiscale by seamlessly linking length scales ranging from the subnano to the macro. Our model accounts for the nonlinear effects in the core of the nanostructure, and it provides a fast algorithm for modeling a large crystallite. It takes only a few CPU seconds to calculate the LSGF for a million-atom model on a standard 3 GHz desktop.

Contributors and Collaborators

R.R. Keller, B. Yang (Materials Reliability Division, NIST); E. Pan (Akron University)

Micrometer-Scale Reliability: Molecular Dynamics

It is widely anticipated that applications of nanomaterials will enable major advances in technology in a variety of fields, including sensors, high-strength materials, medicine, and others. Computer simulations seem to offer a path to quantitative understanding of the behavior of nanoscale materials. The form and parameters of the interatomic forces may turn out to be the most concise and useful representation of materials measurement results. Atomistic simulations are widely used to interpret nanoscale phenomena at the boundary between mechanics and chemistry and to support the plausibility of proposed devices. We are developing the capability to use molecular dynamics simulations to interpret our own measurements, and to assess the accuracy of proposed interatomic potentials.

David T. Read

Various approaches to simulating atomic interactions have been reported. The **first principles** approach uses *quantum mechanical* models of nuclei and electrons. Even though this approach typically includes only the valence electrons, it can only handle a few tens of atoms. Recently, in an innovative study, the first principles approach was used to search for new superalloys.

The **molecular mechanics** approach, widely used for organic molecules such as proteins, requires explicit assumptions about atomic bonding, specifically, a list of which atoms are bonded to which other atoms. The bonds are represented by force laws, and the configuration of complex molecules can be studied. Different force laws may be used for certain types of atoms, depending on the chemical environment. For instance, the carbon–carbon force law in diamond may be different from that in graphite.

Molecular dynamics (MD) treats atoms or molecules as particles that follow *Newton's laws* of mechanics. The particles interact with a prescribed force law, which depends only on the chemical identity of the interacting atoms. The force laws are derived empirically, and the parameters are selected by fitting to measured properties, such as the elastic constants, the vacancy energy, the energy of sublimation, phonon frequencies, and others. This approach can treat solids, liquids, or gases, and can model melting temperatures and equilibrium crystal structures.

Figure 1: *Scanning electron microscope image of the surface of a copper electrodeposit at a magnification of 1 million.*

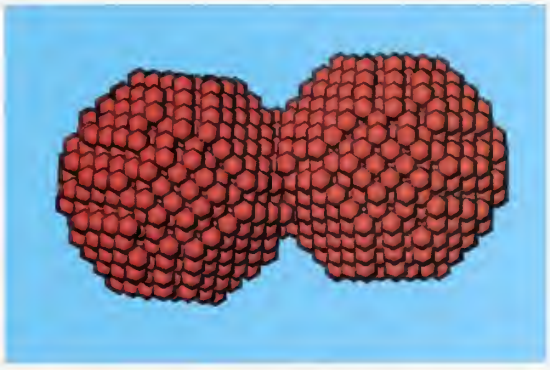

Figure 2: *MD simulation of agglomerated spheres of copper atoms, but with many fewer atoms than the actual structures.*

The many-body potential, introduced in the 1980s, provided some key triumphs of physical insight into the behavior of metals. It allowed quantitative treatment of surface reconstruction, observed in some pure metals under controlled conditions; it allowed understanding of the Cauchy inequality among the elastic constants of metals; and it allowed reasonable values of the energy of a vacancy. Starting in the latter 1980s, Tersoff introduced his potentials for modeling silicon. His innovation was to introduce the dependence of energy on bond angles into molecular dynamics. Even now, MD potentials have been introduced for only a small fraction of possible combinations of atoms. Generally, potentials are available for important and commonly used materials, but not for exotic combinations.

Contributors and Collaborators

J. Rifkin (University of Connecticut; author of MD program used)

Micrometer-Scale Reliability: Solder Reliability

Because of the environmental hazards of lead, the electronics industry is replacing lead–tin eutectic solders with lead-free solders. In doing so, they have created a need for material properties data of the new lead-free solder compositions. Efforts have been made to standardize the collection of the data and disseminate it. A test method has been developed to measure the mechanical properties that uses specimens on the same size scale as that of the solder structures used in industry.

Timothy P. Quinn

Background

Industry groups have pointed out the need for material property data for the new lead free compositions of solder which includes pooling the data that already exists and filling the critical holes in the data especially at low strain rates.

Most of the data that are available for all solders come from specimens that are very large (on the order of millimeters) compared to the solder structures themselves (on the order of hundreds of microns). As the solder structures (solder balls for flip chip packages, for example) become smaller (~150 mm), their dimensions approach the dimensions of the phases in the solder itself. The assumption of a homogeneous material used to analyze the stresses in the structure is challenged. To study this interaction, we have therefore started testing samples that are on the same size scale as current solder structures.

Accomplishments

The long history in the use of current lead-based solders means that these data sets are quite complete and widely available. The modelers and production engineers need equally complete sets of data on the various lead-free alternatives, so they can make informed decisions for their production applications. Researchers are rapidly developing corresponding data on lead-free alloys, but the data are widely distributed among the various technical journals and proceedings. We assisted the National Electronics Manufacturing Initiative (NEMI) efforts by developing a database of properties on the three most common lead-free compositions. Once we had a reasonable base, we published the data on the NIST website, and requested additional data.

The pace of change in the database has slowed, but we have made a long-term commitment to maintain the database on the Materials Reliability Division's Website at: **http://www.boulder.nist.gov/div853/**. We continue to publicize the availability of the database at national technical meetings.

Different test procedures (*e.g.*, loading rates and dwell times) make some of the data inconsistent from laboratory to laboratory and so decrease the accuracy of the models. A Recommended Practice Guide (NIST SP 960-8) was written that recommends standardized procedures to facilitate the comparison of mechanical-property data within the electronics industry.

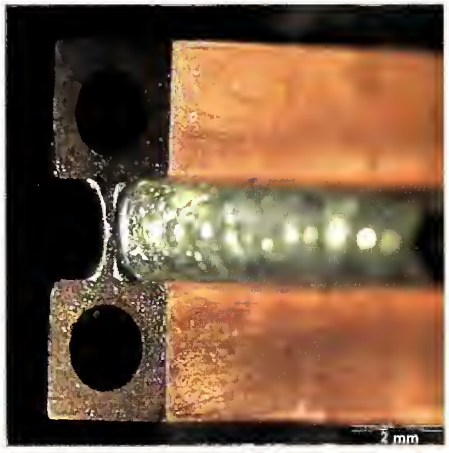

Figure 1: *Solder is cast between two copper blocks to make a "loaf" from which specimens are sliced off.*

A test method that used "miniature" specimens was developed to mimic the size of typical soldered joints. The purpose was to examine the effects of the size of the specimens (and hence the typical structures found in industry) and to fill in the gaps in the data for the lead-free solders. Solder is cast in a Ti mold between two copper blocks (Figure 1), and 200 μm specimens are cut from the blocks. The gauge length of the specimens is 300 μm. We can consistently make specimens with a known thermal cycle and maintain well-defined microstructure. Because the samples are sliced from a relatively large "loaf," a large number of samples can be made in a short time.

Contributors and Collaborators

Yair Rosenthal, Tom Siewert (Materials Reliability Division, NIST); Carol Handwerker (Metallurgy Division, NIST)

Materials Property Measurements

This program responds both to the Materials Science and Engineering Laboratory (MSEL) customer requests and to the Department of Commerce 2005 Strategic Goal of "providing the information and framework to enable the economy to operate efficiently and equitably." For example, manufacturers and their suppliers need to agree on how material properties should be measured. Equally important, engineering design depends on accurate property data for the materials that are used.

The MSEL Materials Property Measurement Program works toward solutions to measurement problems on scales ranging from the macro to the nano, in four of the Laboratory's Divisions (Ceramics, Materials Reliability, Metallurgy, and Polymers). The scope of its activities ranges from the development and innovative use of state-of-the-art measurement systems, to leadership in the development of standardized test procedures and traceability protocols, to the development and certification of Standard Reference Materials (SRMs). A wide range of materials is being studied, including polymers, ceramics, metals, and thin films (whose physical and mechanical properties differ widely from the handbook values for their bulk properties).

Projects are directed toward innovative new measurement techniques. These include:

- Measurement of the elastic, electric, magnetic, and thermal properties of thin films and nanostructures (Materials Reliability Division);
- Alternative strength test methods for ceramics, including cylindrical flexure strength and diametral compression (Ceramics Division); and

The MSEL Materials Property Measurement Program is also contributing to the development of test method standards through committee leadership roles in standards development organizations such as the American Society for Testing of Materials (ASTM) and the International Standards Organization (ISO). In many cases, industry also depends on measurements that can be traced to NIST Standard Reference Materials (SRMs). This program generates the following SRMs for several quite different types of measurements:

- Charpy impact machine verification (Materials Reliability Division);
- Hardness standardization of metallic materials (Metallurgy Division); and
- Hardness standardization and fracture toughness of ceramic materials (Ceramics Division).

Supporting the Materials Property Measurements Program is a modeling and simulation effort to connect microstructure with properties. The Object-Oriented Finite-Element (OOF) software developed at NIST is being used widely in diverse communities for material microstructural design and property analysis at the microstructural level.

In addition to the activities above, all four divisions provide assistance to various government agencies on homeland security and infrastructure issues. Projects include assessing the performance of structural steels as part of the NIST World Trade Center Investigation, advising the Bureau of Reclamation on metallurgical issues involving pipelines and dams, advising the Department of the Interior on the structural integrity of the U.S.S. Arizona Memorial, advising the U.S. Customs Service on materials specifications for ceramics, and advising the Architect of the Capitol on repair procedures for cracks in the outer skin of the Capitol Dome.

Contact: Thomas A. Siewert

World Trade Center: Analysis of Structural Steel in the World Trade Center

In 2002, NIST became the lead agency in a planned investigation of the World Trade Center collapse. The investigation addresses many aspects of the catastrophe, from occupant egress to factors affecting how long the Twin Towers stood after being hit by the airplanes, with a goal of gaining valuable information for the future. A critical aspect of the investigation is the metallurgical and mechanical analyses of structural steels from the Twin Towers and WTC 7. The analyses include characterization of properties, failure modes, and temperature excursions seen by the steel.

David McColskey, Christopher McCowan, and Thomas Siewert

The collapse of the twin World Trade Center Towers on September 11, 2001 was the worst building disaster in human history. Engineers, emergency responders, and the nation were largely unprepared for such a catastrophe. The disaster highlights the following national needs:

- To establish the probable technical causes of the collapses and derive the lessons to be learned;
- To develop and disseminate immediate guidance and tools to assess and reduce future vulnerabilities; and
- To produce the technical basis upon which cost-effective changes to national practices and standards can be developed.

NIST has prepared a technical plan to address these issues (see **http://wtc.nist.gov/**). A primary objective of the investigation is to determine why and how the towers collapsed after the initial impact of the aircraft.

Figure 1: *Instrumentation adjustment on high-temperature specimen.*

As part of this investigation, the Materials Reliability and Metallurgy Divisions in MSEL are studying more than 200 structural steel pieces from the WTC site. Progress in this study is outlined here.

Task 1: Collect and catalog physical evidence. More than 200 pieces of the World Trade Center towers have been collected and brought to NIST. The locations of 41 exterior column panels within the WTC towers have been positively identified, of which 15 were located in or near the impact zones of the aircraft. Specified strengths of structural components have been identified through engineering drawings for the buildings.

Task 2: Failure mechanisms based on visual evidence. Recovered steel is being examined and documented as to failure mechanisms.

Task 3: Property data to support studies of structure performance and airplane impact modeling. Fourteen grades of steel were specified in the design of the WTC towers. All grades have been characterized for room-temperature mechanical properties, and initial high-temperature test results are complete. Testing at high-strain rate is underway to determine the effects of strain rate on the mechanical properties of the outer columns, the inner columns, and the spandrels. Chemical composition and metallographic examinations have been completed on the majority of the steels. Creep, or time-temperature-dependent behavior of some steels will be studied after the high-temperature properties are developed.

Task 4: Correlate determined steel properties with specified properties. As data are generated during the testing phase of the investigation, measured values of strength are compared to those specified in the engineering drawings.

Task 5: Metallographic analysis of steel to estimate temperature extremes. Microscopic, macroscopic and metallographic analyses are under way to determine the maximum temperature excursions seen by the steel.

An interim report containing data generated to date will be released to aid in the modeling of the aircraft impact and building response to subsequent fires. A final report will be complete at the end of FY04.

Contributors and Collaborators

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Infrastructure Reliability: Waveform-Based Acoustic Emission

The major project objective is to develop the scientific underpinnings necessary to enhance acoustic emission (AE) technology through increased, high-sensitivity bandwidth. Current secondary objectives include: (1) developing for many users the missing element of modeling AE signals for multiple sources in specimens with and without edge reflections; and (2) developing rational approaches to analyze AE waveforms to solve the real-world problems of reliable identification and location of sources of AE signals.

Thomas Siewert and Marvin Hamstad

Technical Description

Acoustic emission (AE) refers to the generation of propagating elastic displacement waves as a result of microsized transient energy releases in a material. Monitoring these waves can provide fundamental information about the location and mechanism(s) of the transient-energy release as well as the time/stress history of such releases. The technical approach, which is beyond that currently commercially offered for either resonant or waveform-based AE technology, is to develop the key components relevant to a wideband application of AE technology. These include development of wideband high sensitivity sensor/preamplifiers; high-speed digital recording data-gathering systems of wide dynamic range; finite-element modeling to predict near- and far-field displacement waves from AE sources; wideband experimental AE displacement waveforms from sources in materials of interest; signal-processing techniques to accurately identify source types and their locations; and experimental characterizations of simulated AE wave propagation. The scope in FY03 covered two related phases: (1) detailed analysis of wavelet-transform (WT) results from finite-element modeled AE signals for three key AE source types located at different depths (relative to a plate's top surface) and radiation angles; and (2) use of the WT results to develop an approach to identify the AE source mechanisms.

Accomplishments

Extensive examination of a large WT database revealed that three key frequencies and their associated fundamental modes were sufficient to characterize the most energetic portions of the AE signals. The frequency and Lamb-mode combinations identified were S_0 @ 522 kHz, S_0 @ 270 kHz and A_0 @ 60 kHz. The WT database was calculated from finite-element-modeled AE signals for dipole-type sources in an aluminum plate 4.7 mm thick. The plate has transverse dimensions sufficient that plate-edge reflections do not superimpose on direct signal arrivals.

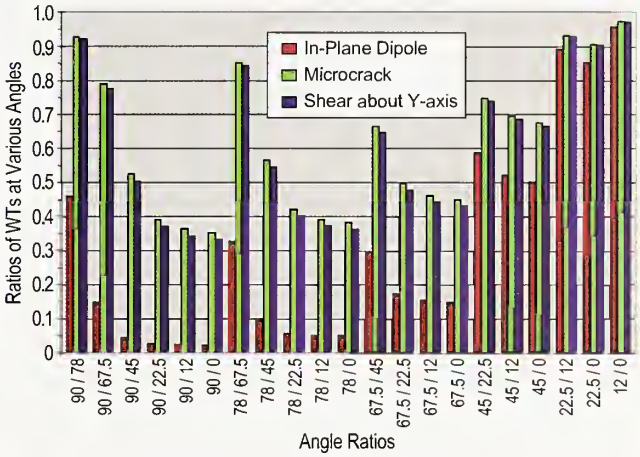

Figure 1: *Ratios at various radiation angles of peak WT magnitudes for the A_0 mode at 60 kHz for all three source types.*

To develop techniques to identify AE source types, detailed analysis of three important source types was carried out. These were an in-plane dipole, a microcrack initiation, and shear without a moment. Important discoveries were made. If the peak WT magnitudes (which represent the arrival of an energetic mode) at each of the three key frequencies were normalized, then the in-plane radiation pattern of these normalized magnitudes for each source type was independent of the depth of the source in the plate. Second, the radiation pattern had differing characteristics for each source type. Figure 1 shows how ratios of the WT peak magnitudes from two different angles can provide a means to distinguish the different source types. In this figure, the in-plane dipole source is clearly distinguished from the other two sources, particularly when one of the angles is 45 degrees or higher. The figure shows angle ratios of the WT peak magnitudes at a propagation distance of 180 mm. Similar figures at the other key frequencies provide additional distinctions between the other sources under study.

Contributors and Collaborators

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Infrastructure Reliability: Charpy Impact Machine Verification

We assist owners of Charpy impact machines in achieving conformance with the requirements of ASTM Standard E 23. We interact with the ASTM Committee responsible for the Charpy impact standard, to improve the service and to maintain a high-quality verification program. We also participate in the activity in ISO Committee TC 164, so our specimens and procedures remain compatible with the associated international and regional standards.

Daniel P. Vigliotti

Technical Description

The Charpy impact test uses a swinging hammer to assess the resistance of a material to brittle fracture. The absorbed energy is measured from a calibrated scale, encoder, and/or an instrumented striker. The low cost and simple configuration of the test have made it a common requirement in codes for materials used in critical structures such as pressure vessels and bridges. This project is handled jointly by the Standard Reference Materials Program (of the Office of Measurement Services), which oversees the administrative aspects of the program, and the Materials Reliability Division, which handles the technical and verification aspects. NIST provides highly characterized standard reference materials (SRMs) to machine owners and independent calibration services, then evaluates the results of tests of these specimens on their impact machines. Owners of machines that meet the requirements of ASTM Standard E 23 are given a letter of conformance, while owners of nonconforming machines are given recommendations on corrective actions. Our special facilities include the three master Charpy impact machines (all roughly 300 J capacity). These three machines are used to establish certified values for the NIST reference materials sold through the Standard Reference Materials Program Office. In addition, we have several more machines (3 J to 400 J capacities) that are used for research purposes.

Accomplishments

We had over 1000 customers for this service in FY03, a gradual increase from the customer base in past years. The great majority of these machines were found to be within tolerances required by ASTM Standard E 23. As usual, many customers took advantage of our support services, as shown by over 2200 emails, 830 faxes, and 3700 phone calls. We immediately contact the machine owner when it fails to meet the verification criteria. In this contact (by phone, mail, email, or fax), we suggest corrective measures. Also, in our laboratory, we tested the 975 specimens necessary to confirm that nine new lots of reference specimens were suitable to enter the SRM inventory.

We are completing a three-year test program that is collecting data on "International Master Batches" of Charpy impact verification specimens. A meaningful harmonization (equivalency) of Charpy V-notch standards around the world is unlikely until the reference materials used for the verification of impact machines in Europe, Japan, and the United States (EN-10045-2, JIS B 7722, and ASTM E 23) share a more common method of certification. The results of this test program will be used to evaluate the use of Master Specimens as a common control in the certification procedure for CVN verification specimens between the three National Measurement Institutes. It will also evaluate machine variables, offsets, uncertainty, and other factors relevant to the harmonization of our respective systems. A major outcome will be the multi-year comparison of the equivalency of the energy scales used to measure absorbed energy by the United States, Europe, and Japan. We are helping to organize another international symposium, "Second Symposium on Pendulum Impact Machines: Procedures and Specimens," to be held in conjunction with the November 2004 meeting of ASTM Committee E 28 in Washington, D.C. Previous symposia have provided valuable insight into improvements in our program.

Chris McCowan serves as the Chairman of ISO TC164 SC4 P on pendulum impact and also as the Chairman of ASTM Subcommittee E28.07 on impact testing. Dan Vigliotti continues as the Chairman of the Task Group that oversees Standard E 23, the main standard for Charpy impact testing.

This past year, we distributed over 1000 copies of a new training video on the operation of the verification program. Initial comments have been quite favorable.

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Organizational Charts

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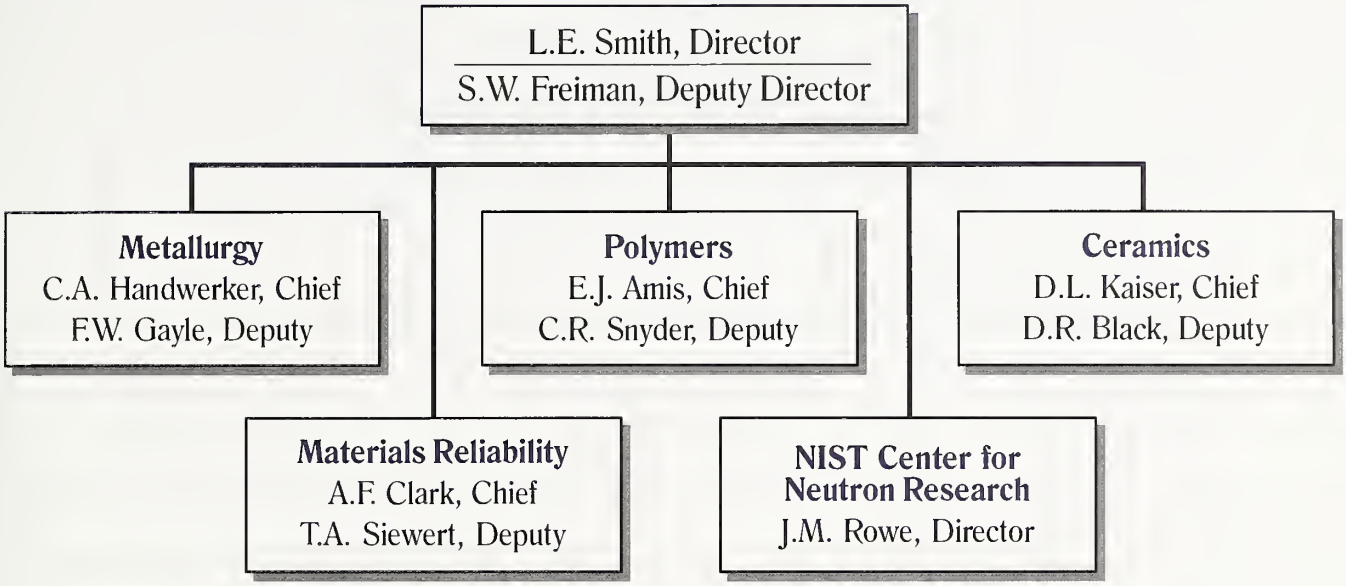

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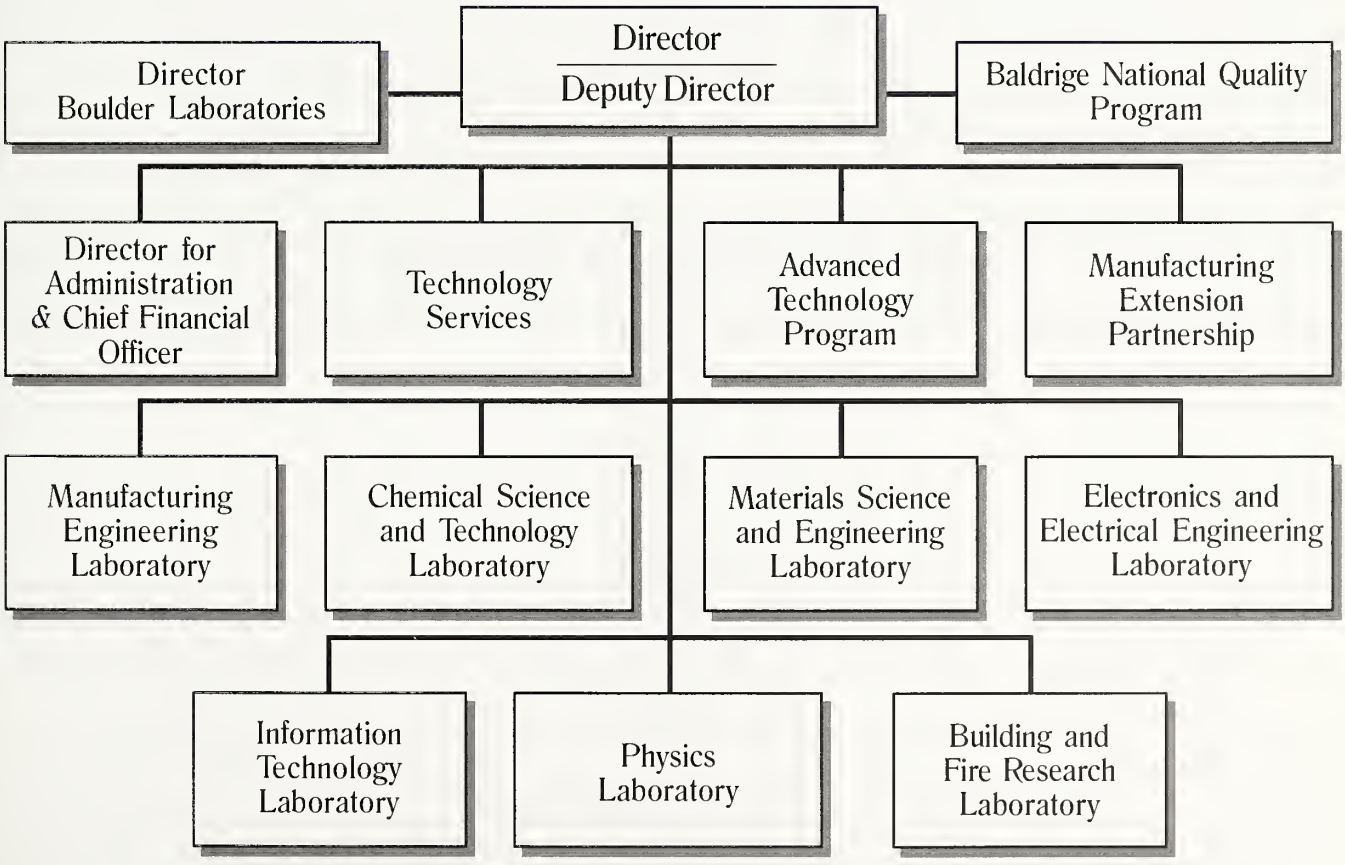
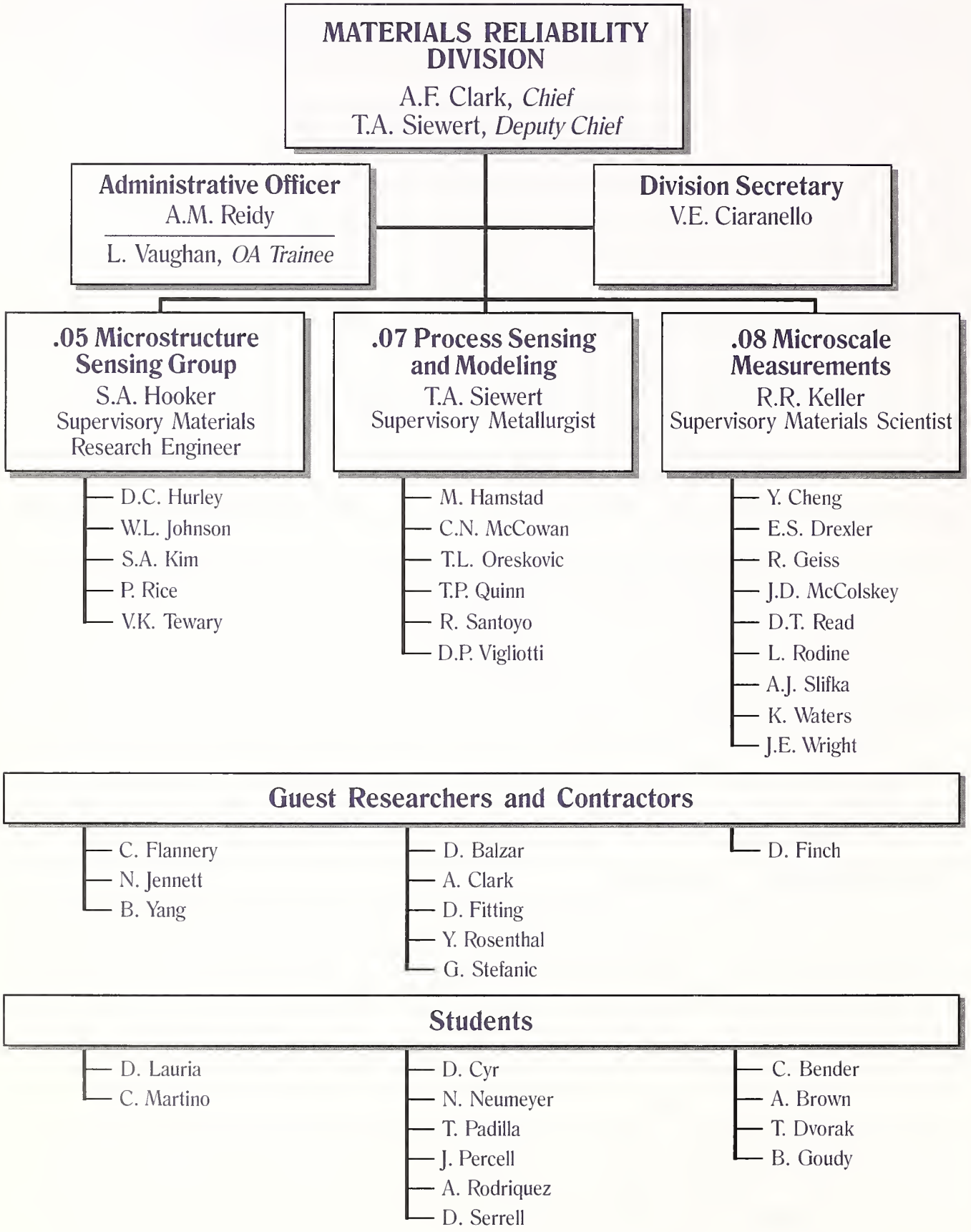


Materials Reliability Division Organization Chart — 2003



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